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The Effects of Ethylamine Decrystallization of Cellulose Fibers on the Viscoelastic Properties of Paper

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THE EFFECTS OF ETHYLAMINE DECRYSTALLIZATION OF CELLULOSE FIBERS ON THE VISCOELASTIC PROPERTIES OF PAPER

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INTRODUCTION AND PRESENTATION OF THE PROBLEM

In the past, considerable time and energy have been spent in attempting to elucidate the mechanisms by which paper responds to stress. In general, paper has been considered a network structure in which relatively strong fibers are held together by relatively weak bonds. On the basis of this hypothesis, it has been suggested that the primary mechanism by which this structure deforms under stress is the rupture of these weak fiber-to-fiber bonds. Rance (1-3) has discussed extensively the evidence to support these views.

Brezinski (4,5), however, proposed, and presented evidence to support, the hypothesis that the deformation is controlled by molecular processes in a manner similar to the behavior of continuous, polymeric materials such as viscose and nylon fibers. More recently, however, investigators have offered evidence that both mechanisms of response, i.e., interfiber and intrafiber, may play a part in determining the over-all behavior of paper under stress (6). It is interesting to note that while Brezinski has been criticized for not recognizing the possible importance of the former mechanism (that is, interfiber), Rance has been criticized for not recognizing the possible importance of the latter.

In view of these controversies, it was felt that more direct evidence was needed to determine the relative importance of the two hypothesized mechanisms of deformation. Consequently, the purpose of this work was to study the relative effects of changes in both interfiber and intrafiber structure on the final response of paper to stress.

APPROACH TO THE PROBLEM

In view of Brezinski's hypothesis (4), it was desirable to examine this molecular point of view more directly by studying the effects of changes in intrafiber structure on the behavior of paper under stress. Then, by making similar studies of the effects of changes in interfiber properties, a comparison could be made of the relative importance of each and how the relationship between them varied as the over-all sheet structure was altered.

One of the most important molecular characteristics of cellulosic fiber structure is the high degree of lateral order or "crystallinity." This type of molecular arrangement, in which certain segments of the large cellulose molecules are bound firmly together by secondary valence forces, produces a fiber structure with considerable strength and durability. Alfrey (7) has discussed extensively the role of crystallinity in the mechanical and viscoelastic behavior of polymeric materials.

Alteration of this crystallinity was therefore chosen as a reasonable means of varying the intrafiber, molecular structure of cellulose fibers. Until fairly recently, however, methods of changing the degree of molecular order have also caused significant changes in such properties as the molecular degree of polymerization and the crystal lattice configuration. In 1952, workers at the Southern Regional Research Laboratories of the U. S. Department of Agriculture patented a process for the treatment of cellulose fibers with various alkylamines (8), resulting in a marked decrease in the degree of molecular order. Of the several amines tested, monoethylamine was the most effective without

simultaneously degrading the fibers or changing their crystal lattice structure, provided proper methods and precautionary procedures were used. A more recent publication (9) demonstrated that different degrees of crystallinity reduction could be achieved easily by treatment of the fibers in varying concentrations of aqueous ethylamine solutions. Consequently, this procedure was chosen as a reasonable means of varying the molecular structure of the fibers to be used in this study.

The comparative portion of the investigation, i.e., the study of the relative effects of interfiber variations on the viscoelastic properties, was accomplished by altering the fiber-to-fiber bonding in the paper by a wet-pressing technique. This provided an effective means of changing the sheet macrostructure without affecting the intrafiber structure.

The viscoelastic properties of the resultant handsheets, varying in both intrafiber and interfiber properties, were studied by means of tensile creep tests. The techniques and equipment used were similar to those employed by Brezinski (4). This procedure, as well as being relatively simple to carry out, allows a separation of the variables of stress, time, and strain. This is contrasted to the complex time sequences of stress or strain which are encountered in a typical load-elongation test, with the resultant difficulties of interpretation.

HISTORICAL REVIEW OF THE MECHANICAL AND VISCOELASTIC PROPERTIES OF PAPER

Some of the first studies of the mechanical and viscoelastic properties of paper were conducted by Gibbon (10) and Farebrother (11). These workers reported extensive stress-strain data on paper and board and concluded that irreversible deformations caused by tensile loadings were the result of fiber slippage.

Steenberg, et al. published a series of papers by Swedish workers dealing with the load-elongation properties of a number of different types of paper (12-16). A significant contribution was made when they demonstrated the dependence of observed mechanical behavior on such variables as rate of straining or loading, temperature, relative humidity, specimen orientation with respect to the paper machine, and previous mechanical history.

Steenberg (13) introduced the concept of "microcreping" to explain the irreversible deformations which had been observed. It was envisioned that there exists in the unstrained fibers a superficially invisible creping or kinking which is removed when the fibers are loaded. Rance (3) has criticized this hypothesis, contending that since most papers are several fiber layers thick, such a randomly bonded, three-dimensional structure would undergo considerable fiber-to-fiber bond rupture before such superficial creping would be removed. In a later paper, Steenberg (16) amended his concept of microcreping to include the interesting belief that all deformations of paper must be considered ultimately reversible since they cannot be proved permanent.

Rance (1-3, 17), after rejecting the concept of microcreping, suggested that nonrecoverable deformations of paper are the result of the rupture of fiber-to-fiber bonds at an increasing rate until the test is terminated by rupture of the specimen. The time-dependent component of the deformation was attributed to a combination of elastic elongation of fibrous elements, plus the frictional sliding of fibers past one another. Some support was given to Rance's theories by the work of Maynard (18) who found that paper subjected to straining in the post yield region of load-elongation tests increased in thickness as much as 10%. He concluded that the rupture of fiber-to-fiber bonds was responsible for this phenomenon. Sanborn (19), however, reported a decrease (to a constant value) in the thickness of specimens subjected to tensile stress and concluded that this behavior was due to the removal of cockle.

McKensie (20) presented the load-elongation curves of a number of different types of paper as a function of beating time. He noted that at relatively high beating levels, the initial and post-yield portions of the load-elongation curves of paper prepared from different pulps tended to become constant. He concluded that such relatively strong papers behaved as hydrogen-bonded continua and that the only explanation of the irreversibility of the straining process is that the rupture of hydrogen bonds is taking place.

More direct evidence to support the theory of interfiber bond rupture was presented by the work of Nordman, Gustafsson, and Olafsson (21-24) who developed a technique for measuring the optical properties of paper

under tension. On the basis of the findings of Haselton (25) and others (26, 27) that the specific scattering coefficient is directly related to the unbonded surface area of paper, these workers measured the changes in the scattering coefficient during load-elongation cycles. These changes were found to be linearly related to the energy absorbed by the paper during the straining. From the slopes of the scattering coefficient-energy loss curves, a so-called bonding strength was calculated which appeared to be unaffected by various sheet-forming variables. However, certain workers (28, 29) have questioned this interpretation of the data because it does not take into account such possible factors as intrafiber light scattering, the separation of unbonded areas, and energy losses due to the straining of individual fibers.

Sanborn (19), while able to confirm Nordman's linear correlation between change in scattering coefficient and irreversible deformation, was not able to obtain a linear relationship between the change in scattering coefficient and energy loss. He found that the latter relationship was affected by the duration of testing and suggested that appreciable amounts of energy might be absorbed by the fiber elements within a sheet during deformation. He pointed out that if this hypothesis is correct, it would invalidate any attempt to calculate a bond strength from light scattering-energy loss data.

The most direct evidence to date that rupture of fiber-to-fiber bonds takes place in paper under stress was recently published by Page, et al. (30-32). These workers developed a microscopic technique for observing interfiber areas of paper which were in optical contact while

the sheet was in tension. Their data indicated that some bonds broke very quickly while the areas of others seemed to decrease slowly during the deformation. One of their most important observations, however, was that bond areas tended to break partially rather than completely, and that when total breakage did occur, it was confined primarily to the smaller bond areas. Examination of sulfite handsheets, prepared from pulps beaten 0, 10, and 30 minutes, revealed that almost one-half the bond areas remained unbroken, about 45% were ruptured to varying degrees, and only 6% broke completely.

In addition, Page, et al. (32) found the frequency of bonds per fiber to be quite high, and concluded that the role of friction in simulating viscoelastic behavior, as suggested by Rance (17), must be completely discounted, since virtually every site at which it could operate is bonded. A similar opinion has been expressed by Van den Akker (33).

In 1955, Brezinski (4, 5) published the results of an extensive study of the creep and recovery behavior of alpha-pulp handsheets. He observed a degree of ideality in this behavior which, he suggested, would not be expected if macroscopic changes in sheet structure made significant contributions to the total delayed deformation. He noted the similarities between his results and those reported by such workers as Press (34), Mark and Press (35), and Leaderman (36) in their studies of textile materials, and concluded that molecular mechanisms control the manner in which paper responds to stress. Creep deformations at early times were attributed to configurational changes in the amorphous

regions of the fibers and those at later times were related to reversible and irreversible changes of phase. In addition, Brezinski found that a considerable portion of the so-called nonrecoverable creep of paper could, in fact, be recovered by subjecting the specimen to a humidity during recovery which was higher than that used during creep. This was compared to similar behavior observed with viscose by Mark and Press (35).

Ivarsson (15) investigated the mechanical conditioning of paper by subjecting specimens to several load-elongation cycles. He found that many cycles were needed before the specimens approached reversibility, although a large amount of the irreversible deformation was removed in the first cycle. He suggested that this behavior was related to a reorganization of crystalline regions within the fibers, rather than to a phase change to a more crystalline state as observed in rubber (37).

Schulz (38) studied the mechanical properties of papers which had been subjected to various degrees of straining during the drying period. He found that such "wet-straining" markedly affected the mechanical and creep-recovery behavior of the sheets. As the degree of wet-straining was increased, such factors as tensile strength and elastic modulus (in the direction of straining) increased and the creep and recovery response was significantly reduced. Schulz suggested that this behavior was caused by a redistribution of the elements which contributed to the support of the load, increased straining increasing the number of load-carrying components in the sheet.

Work presented recently by Van den Akker, et al. (6) lends support to the hypothesis that both interfiber and intrafiber mechanisms play

a part in determining the over-all response of paper to stress. These investigators measured the number of dyed fibers which were broken at or pulled intact from the zone of rupture of a tensile test. They found that even in relatively weak sheets, the percentage of fibers broken was substantial, and that as the strength of the paper increased, this percentage also increased.

A number of workers have attempted to analyze the structure of paper and to relate its mechanical properties to those of its constituent fibers. Onogi and Sasagari (39) have done this for papers strained in the elastic region, deriving expressions for the density, Poisson's ratio, elastic modulus, and dynamic viscosity of paper from the density, elastic modulus, radius, segment length, and angular distribution of the fibers within it. However, the value of this presentation is seriously limited by the erroneous assumption that the forces acting on all the fibrous elements are parallel to the direction of the externally applied load (40).

A comprehensive theoretical study of this nature has been presented recently by Van den Akker (40) in which the analysis is divided into considerations of the "elastic" and "plastic" regimes of paper response. Expressions are developed, for the former region, for such properties as Young's modulus, Poisson's ratio, and the modulus of rigidity. For the latter region, the theory deals with expressions related to "torque" and "tension" types of bond failure.

Extensive surveys and discussions of the pertinent literature in this field have been presented by Rance (3) and Brezinski (4). More

recent developments and discussions, including work done up to 1961, may be found in the proceedings of the symposium on the Formation and Structure of Paper held in Oxford, England in September, 1961 (41).

PRELIMINARY CONSIDERATIONS

THE STRUCTURE AND PROPERTIES OF CELLULOSIC FIBERS

The following brief discussion is presented to familiarize the reader with some of the more important structural and behavioral characteristics of cellulose and cellulose fibers, basic to an understanding of this work and of the problems involved. Because of the nature of the material used in this study, emphasis will be placed on the properties of cotton fibers. Quite comprehensive discussions of the properties of cellulosic materials in particular may be found in Ott and Spurlin (42) and of polymeric materials in general, in Alfrey (7). This discussion is taken largely from Howsmon and Sisson (43).

MOLECULAR STRUCTURE

Cellulose is a polymeric substance composed of macromolecules of anhydroglucose units which are held together by beta-1,4 glycosidic bonds. The natural celluloses have a degree of polymerization of several thousand, with some chains, of course, being considerably longer (44). The values obtained will depend upon the source of the material and the method of determination.

Cellulosic fibers are generally highly crystalline structures and exhibit many of the properties of other polycrystalline materials. The tendency for cellulose molecules to crystallize is enhanced by the rigidity of the anhydroglucose rings and the strong attractive forces

associated with the hydrogen bonding of the hydroxyl groups. In addition, the flat shape of the rings and the spatial distribution of the hydroxyl groups provide a highly symmetrical configuration which is easily fitted into a crystal lattice.

All natural celluloses, with the possible exception of one, have a crystalline structure which has been designated as cellulose I. The generally accepted lattice structure of the cellulose I unit cell was first proposed by Meyer and co-workers (45). They suggested unit cell dimensions of a = 8.35 A., b = 10.3 A., c = 7.9 A., and $\beta = 84°$. The planes of the anhydroglucose units lie in the ab plane and the axes of the cellobiose units are parallel to the b axis. The angle β is that between the ab and bc planes. It should be noted that because of the absence of single cellulose crystals, the exact definition of the unit cell is impossible. All such unit cell dimensions are approximations which are dependent upon lattice distortions in the polycrystalline aggregates (43).

When cellulose is precipitated from solution or regenerated from some addition compound of cellulose I, such as the alkali cellulose formed in mercerization, a lattice configuration differing significantly from that of cellulose I may be obtained. This structure is generally referred to as cellulose II. The molecules are displaced relative to one another along the a axis and are rotated about 30° out of the ab plane. The generally accepted dimensions were found by Andress (46) to be: a = 8.14 A., b = 10.3 A., c = 9.14 A., and $\beta = 62°$.

The third major lattice configuration of cellulose may be obtained by decomposition of the swelling compound of cellulose with liquid ammonia. This structure has been designated as cellulose III and its dimensions and molecular arrangements are similar to those of cellulose II. Legrand (47) has proposed dimensions of: $\underline{a}$ = 7.74 A., $\underline{b}$ = 10.3 A., and $\underline{c}$ = 9.96 A. with $\beta = 58°$. Others have proposed different unit cells. On the basis of present evidence, it is impossible to establish any structure conclusively.

The marked effects of treatments like mercerization on such fiber properties as sorption, swelling, and tenacity have been known almost since the discovery of the processes. When evidence was first obtained that these procedures also changed the crystal structure, there was a tendency to attribute great significance to the effect of this transformation on the physical properties. However, subsequent evidence has shown that the effects are more probably related to such factors as the accompanying decreases in amount of crystalline material and changes in crystallite orientation. Thus, as Howsmon and Sisson point out, it seems more correct to regard the various transformations from one lattice structure to another as interesting but accessory phenomena, for none of the differences in important technological properties can be attributed solely to differences between crystal forms (43).

In general, there have been three main theories concerning the nature of the crystalline-noncrystalline multiphase structure of cellulose fibers. They will be reviewed only briefly here.

The micellar theory was first proposed by Nägeli in 1858 (48). The fibers were considered to be made up of discrete, anisotropic crystalline particles, much as a wall is made up of bricks, held together by an amorphous cementing material. However, this theory was abandoned when ultracentrifuge and viscosity data indicated that the cellulose molecule was of the order of ten times longer than the estimated length of the micelle.

The continuous structure theory was suggested by such investigators as Sponsler (49) and Astbury (50) who regarded the structure as a molecular order which only approximated lattice perfection and which was marked by numerous local imperfections.

A consolidation of the best points of both the micellar and continuous structure theories led to the development of the fringe micellar theory. The micelles are considered as statistically distributed regions of latticed order in a mass of substance consisting of approximately parallel chain molecules. There are no sharply defined crystalline boundaries but only gradual transitions from regions of high lateral order to regions of low lateral order. The crystalline areas in cellulose may vary in size, shape, and degree of perfection, with an infinite variety of distributions of size, shape, and degree of perfection possible (43).

In the course of this dissertation, the terms "crystallinity" and "degree of crystallinity" will be used frequently. However, a belief that such absolute states as purely crystalline or purely amorphous regions exist in cellulose fibers is not implied. It is recognized that the molecular structure of cellulose exists in a state of widely varying and complex

distributions of lateral order, and that any attempts to label particular cellulose materials with a "degree" or "percentage" of crystallinity must be viewed with caution and with the various limitations in mind. These terms are employed merely for convenience and because of their general usage in this field.

THE MACROSTRUCTURE OF CELLULOSE FIBERS

Morphologically, native cellulose fibers consist of a thin outer membrane or primary wall and a thicker secondary wall. The majority of the crystalline cellulose exists in the secondary wall which, under the optical microscope, appears to be made up of layers of stringlike particles called fibrils. These fibrils lie side by side and have diameters of the order of tenths of a micron. Generally, they are not arranged perfectly parallel to the fiber axis but are inclined at an angle which may vary in different fibers and even in different layers of the same fiber. Often, the direction of spiral is different in different layers of the same structure. In cotton, the fibrils may change their direction of spiral at frequent intervals along the length of the fiber.

The fibrils are in turn made up of microfibrils which are generally considered to be about 100 to 150 A. in diameter and of indeterminate length (51). The microfibrils may then be composed of bundles of finer units called micelle strings as proposed by Frey-Wyssling (52). He suggests that these are in turn built up of the crystalline and disordered regions of the basic molecular structure.

MECHANICAL PROPERTIES

Native cellulose fibers exhibit many of the mechanical properties generally associated with oriented, crystalline polymers: for example, relatively high tensile strength and modulus of elasticity, and relatively low elongation. These properties, of course, may be markedly affected by the morphological characteristics of the fibers involved. For example, Wakeham and Spicer (53) have suggested that the regions of fibrillar reversal in cotton fibers act as weak links when under tensile stress.

Most studies of the mechanical properties of cotton fibers have been carried out with the fibers in the form of yarns, fabrics, or even paper. However, Wakeham (54) reports a load-elongation curve for a dry cotton fiber which, he suggests, is characteristic of many cellulosic materials. There is an initial region of high extensibility in which the kinkiness of the fiber is removed. This is followed by a relatively elastic region above which there is definite indication of yield. At higher stresses, the curve may turn slightly upward along the load axis indicating structural alterations leading to reinforcement. Just before rupture, the curve often turns away from the load axis due to deterioration of the fiber structure. Different fiber samples may, of course, show appreciably different behavior, such as complete absence of a reinforcement region.

Sisson (55) and Sisson and Clark (56) have shown the strength of fibers to be highly dependent upon fibrillar orientation in the cell wall. As the fibrils become oriented more along the fiber axis, the tensile strength increases. Orr, et al. (57) have postulated that much of the elongation of cotton fibers is related to the reversals of the spiral

structure of the fibers. Unlike yarn, in which the twist is entirely in one direction, these reversals allow the structure to untwist when load is applied. These workers report the observation of such behavior in fibers under tensile load.

Staudinger and Jurisch (58) studied the relation between tensile strength and cellulose degree of polymerization (D.P.) by treating cotton with acids to form a homologous series. The strength increased with an increase in D.P. from about 200 to 700, increasing only slightly then from a D.P. of about 700 to 1000. Above this level, the strength was essentially unaffected by changes in the D.P. Workers have suggested that this lack of dependence of strength on D.P. at high degrees of polymerization is related to the fact that the lengths of the larger molecules are considerably greater than the lengths of the crystallite regions (43).

The effects of changes in the extent of crystallinity on strength properties have been limited primarily to investigation of yarns. In many cases these effects have been complicated by variations in other properties such as crystallite orientation and gross morphological structure. Ziifle, et al. (59) have shown that mercerization, which reduces the crystallinity, will decrease yarn tenacity and increase stretch if the process is conducted with the yarn in a relaxed condition. However, if tension is used during treatment, strength increases and elongation decreases.

Somewhat more reliable results may be obtained when crystallinities are altered by treatment in ethylamine, since this process has been shown

to have little effect, under certain conditions, on crystal structure, crystallite orientation, and degree of polymerization (8, 9, 60-63). Ziifle, et al. (59) reported an increase in tenacity of cotton yarns treated in ethylamine. Similar data were reported by Susich (64). However, Tripp, et al. (65) reported a decrease with treatment in the amine.

More complete discussions of the mechanical properties of fibrous materials may be found in Alfrey (7), Leaderman (36), and Ott and Spurlin (42).

CHEMICAL REDUCTION OF CRYSTALLINITY

The reduction of the degree of crystallinity in cellulose fibers by chemical, as well as physical, means has been known for some time. So-called amorphous cellulose may be prepared by evaporation of ammonia from ammonia cellulose (66), by denitration of cellulose nitrate (67), and by dry grinding of cellulose in a vibratory ball mill (68). The reduction in crystallinity accompanying mercerization of cellulose is well known.

One of the most important methods for the alteration of cellulose lateral order structure involves the use of various alkylamines as strong swelling agents (8). Of these, monoethylamine appears to be the most effective for decrystallization in a single treatment. Pretreatment in ethylamine, followed by treatment in one of the higher amines, such as hexylamine, however, does result in more complete decrystallization.

When treating with ethylamine, maximum effect is obtained with the use of the anhydrous chemical (9). The crystalline structure remaining depends upon the procedure of amine removal. If the reagent is removed by evaporation, the resulting cellulose contains the cellulose III lattice; if it is removed by washing with water, cellulose I remains. Other procedures and combinations of procedures may be used yielding various mixtures of the two configurations (62, 63). The extent of decrystallization with the anhydrous amines depends upon temperature and time of treatment (8).

Loeb and Segal (9) have shown that the extent of crystallinity reduction may also be controlled by treating the cellulose in various concentrations of aqueous ethylamine solutions. They found that the crystallinity, as determined by acid hydrolysis, was reduced gradually from 91 to 26% by treating in aqueous solutions ranging in concentration of ethylamine from 70 to 90%. Appreciable degrees of decrystallization are not obtained in concentrations much lower than 71% ethylamine. This concentration corresponds to a one-to-one mole ratio of amine and water. These workers have suggested that an excess of amine is necessary before penetration into the crystalline regions of the cellulose is possible, because of the strong complexing tendencies of the amine and water. The bulk of the reduction takes place during the first few minutes of treatment and then continues gradually until the maximum effect is reached in about four hours (61).

Further investigations by these workers have shown that treatment in ethylamine produces no change in crystallite orientation (61) and,

if proper precautions are taken to exclude air from the reaction, no appreciable reduction in degree of polymerization is observed.

MEASUREMENT OF THE DEGREE OF FIBER CRYSTALLINITY

In general, methods used for the determination of the fiber "degree of crystallinity" may be classified into three types. First, there are chemical methods in which the accessible hydroxyl groups react with a reagent unable to penetrate the more ordered regions of the fiber. Esterification with formic acid is an example of this type of determination (69).

Second, the kinetics of certain reactions with cellulose may be studied, in which the reaction is divided into a period of initially rapid rate followed by a period of slower rate. By assuming that the initial rapid rate involves reaction in the amorphous regions, extrapolation of the slow-rate stage will give a measure of the amount of intercrystalline material present. The most common method utilizing this principle is the determination of rates of hydrolysis in hydrochloric acid solutions. However, there is evidence that the amount of crystalline cellulose determined in this manner is affected by a rapid recrystallization of cellulose which accompanies chain cleavage in aqueous media (70).

Third, the various physical properties of cellulose may be studied, based on the fact that crystalline cellulose will have different properties than noncrystalline cellulose. For example, material with crystalline structure will exhibit better x-ray diffraction, higher density, and lower moisture regain than less ordered material.

It must be emphasized that while many of these methods give so-called "absolute" crystallinity measurements, the results do not generally agree, although most tend to rank different materials in the same order. These differences, for the most part, can be related to uncertainties in the methods themselves and to certain assumptions involved in their interpretation. Care must be taken in drawing conclusions about sample structure and properties and in comparing results obtained with different methods.

Because of the nature of the cellulose crystalline structure and the limitations of chemical methods of characterizing it, it has become more and more acceptable in recent years to use the terms "accessible" and "nonaccessible" rather than noncrystalline and crystalline. Whereas the latter terms are somewhat ambiguous, the former denote specific experimental quantities which are definable under the conditions used in their determination. Whether they are more capable of characterizing the structure of cellulose materials, as compared to any other methods, however, is questionable.

The first quantitative procedures for evaluating cellulose structure by x-ray diffraction techniques were reported by Hermans (71) and Kast and Flaschner (72) in 1948. Both methods define crystalline areas as those which contribute to the intensity maxima of the three main diffraction peaks of cellulose--the 101, $10\bar{1}$, and 002 reinforcements. These workers arrived at a crystalline content by comparison of the integrated intensities of the maxima and the intensity of the background scattering from the amorphous regions. As with most of the x-ray techniques, these

methods are dependent upon a somewhat arbitrary estimation of the influence of the so-called amorphous regions.

Numerous other empirical methods of relating the various areas, maxima, minima, and half-widths of the x-ray diffraction peaks of cellulose have been postulated, among them being those of such workers as Conrad and Scroggie (73) and Ant-Wuorinen (74). Recently, Segal, et al. (75) proposed an additional technique which correlated well with determinations by acid hydrolysis, infrared analysis, moisture regain, and density. A "Crystallinity Index" was defined in which the intensity of the 002 diffraction maximum was compared to the so-called amorphous intensity at a Bragg angle of approximately 18°. This amorphous intensity was determined by studying the patterns of cellulose samples which had been subjected to progressively more severe grinding in a vibratory ball mill. After 240 minutes of grinding, a constant pattern was obtained with a relatively broad maximum at approximately 18° 2θ, the curve sloping off gradually on both sides. This curve was assumed to represent the typical background scatter of the amorphous regions of the fiber structure. Thus, the crystallinity index was defined as

$$\text{Cr.I.} = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

where I_{002} is the intensity of the diffraction from the 002 plane and I_{am} is that of the background scatter measured at $2\theta = 18°$.

It must be emphasized that the values obtained from this index do not imply an absolute percentage of crystalline material in the specimens

examined, but can merely serve to show relative differences between them. It suffers from all the limitations discussed earlier concerning attempts to classify portions of the cellulose structure as either "crystalline" or "amorphous." In addition, Nichols (76) has pointed out that all the x-ray diffraction techniques rest upon the assumption that equal masses of the two regions will exhibit equivalence in scattering power.

However, when only relative differences between or relative ranking of samples are desired, as in this work, the x-ray techniques are quite attractive, considering the ease and convenience of their operation. The method of Segal, et al. (75) is especially attractive in view of the limited calculations involved; no measurements of areas are required.

VISCOELASTICITY AND THE MEASUREMENT OF VISCOELASTIC BEHAVIOR

A viscoelastic body may be defined as one which exhibits both instantaneous elastic deformation and delayed, time-dependent deformation when subjected to an externally applied stress. An ideal viscoelastic material obeys the superposition principle (7).

The superposition principle, as proposed by Boltzman (77) states that a viscoelastic substance will respond to a complicated series of loading and unloading steps as if each load were applied independently for an infinite period of time, and that the deformation at any time will be the simple algebraic sum of the deformations due to all of the loads in the sequence at that instant. In such a consideration, the removal of a load is considered as an application of a negative load;

the principle applies only to reversible, time-dependent deformations. Materials which follow the superposition principle are said to exhibit "linear viscoelastic response" in which the deformation at any time is directly proportional to the applied stress. Leaderman (36) has extended the principle to include materials whose time-dependent deformations are not directly proportional to the applied stress.

In general, tests of viscoelastic behavior may be divided into three categories: (1) sinusoidal techniques, (2) constant stress or strain techniques, and (3) other time-dependent techniques.

In sinusoidal studies, a sinusoidal stress is imposed upon the specimen and the resultant strain, which also varies sinusoidally but usually out of phase with the applied stress, is studied as a function of frequency. Although tests of this type have not often been applied to paper, their use in investigations of other polymeric materials have been discussed by Leaderman (36), Alfrey (7), and Ferry (78).

Constant stress or strain methods generally involve use of one or both of two types of techniques: stress relaxation or creep. In the stress relaxation test, the specimen is held at a constant strain and the decay of the stress necessary to maintain this strain is studied as a function of time. Generally, this procedure is more fundamental than the creep test since no dimensional changes take place during the test. However, it has been found to be dependent upon the manner in which the strain is reached (79) and is often quite difficult experimentally. The creep test, on the other hand, involves merely the application of a constant stress and the measurement of a resultant strain as a function of

time. Such step-function experiments as stress relaxation and creep may be carried out with the sample in tension, torsion, shear, or compression.

Load-elongation tests, which are often applied to paper, are an example of time-dependent techniques in which the stress or strain are varied as some function of time. In such tests, the stress or strain is generally increased at a constant rate with the sample in tension. Most frequently the test is continued until rupture of the specimen, but may be terminated short of that point with the response being studied during cycles of loading and unloading.

While load-elongation tests are quite convenient to carry out, interpretation of the results is made difficult by the relatively complex time sequences involved. This interpretation is further complicated if the specimen under consideration exhibits a relatively large amount of time-dependent deformation. Tests in which either stress or strain is held constant, however, are more easily analyzed. A method of this type, suitable for use in testing paper, is the tensile creep test. This technique is used extensively in this study.

As noted earlier, a viscoelastic material subjected to an applied stress will exhibit both immediate elastic deformation and time-dependent deformation. The time-dependent deformation may in turn be made up of recoverable and nonrecoverable portions.

The immediate elastic deformation is dependent only upon the stress applied and is believed to be the result of the stretching of primary

and secondary bonds and bond angles. It is considered to occur instantaneously upon application of the stress and to be proportional to the stress, but is often quite difficult to measure in the presence of short-time delayed deformations.

The recoverable or delayed elastic deformation is defined as that portion of the time-dependent strain which is recoverable, after removal of the stress, at the test conditions of time, temperature, and relative humidity. It is important that these conditions of recovery be specified clearly since the reversibility of time-dependent deformations may be highly dependent upon such factors as time, temperature, and relative humidity. Delayed elastic behavior is believed to be related principally to configurational changes in molecular structure. These changes are the result of the tendency for polymer molecules to assume statistically preferred configurations under the influence of an external stress. Reversible changes of phase, however, may also contribute a portion of the recoverable deformation.

It follows, then, that the nonrecoverable deformation is that portion of the delayed response which is not recovered, after removal of the stress, at the test conditions of time, temperature, and relative humidity. Nonrecoverable deformation in polymeric materials is often attributed to the movement of entire molecules relative to one another, although such behavior in a highly crystalline structure such as cellulose seems quite unlikely. Some workers have suggested that irreversible crystallization may take place under the influence of stress (4, 36); Leaderman (36) has discussed this possibility in connection with nylon.

Many so-called "permanent" deformations may actually be metastable structures which exist only at the specific conditions used in the test. Leaderman (36) and Mark and Press (35) have shown that considerable portions of the "nonrecoverable" strains of various textile filaments may actually be recovered upon plasticization. These apparently stable states may be related to strong intermolecular forces such as those which arise from the hydroxyl groups in cellulose.

The foregoing discussion of viscoelastic behavior was taken largely from Alfrey (7) and Brezinski (4). The interested reader is referred to these authors for more complete treatments.

EXPERIMENTAL PROCEDURES AND TECHNIQUES

MEASUREMENT OF THE DEGREE OF CRYSTALLINITY

Because of the ease and convenience of the technique, the x-ray diffraction method of Segal, et al. (75) for the determination of degree of crystallinity was chosen for this study. It must be emphasized again that the "Crystallinity Index" defined by these workers can only be used to give a relative ranking of samples. The true relationship between the Index and the actual amount and distribution of molecular order within the tested material is unknown. However, in this study it was felt that it would be sufficient to show merely that differences existed between pulps; therefore, such a relative ranking is satisfactory.

PREPARATION OF PULPS

Samples of pulp to be tested by x-ray diffraction were first air dried, fluffed in a Waring Blendor, and then passed through a small Wiley mill fitted with a 40-mesh screen. A 0.500-g. portion was placed in a one-inch diameter pellet mold, leveled, and pressed at 6000 pounds pressure (7650 p.s.i.) for fifteen seconds (75, 80).

The resulting disk (ca. 0.040 in. thick) was pressed into a one-inch diameter hole in the center of an aluminum mounting plate so that the surface of the disk was flush with the surface of the plate. The dimensions of the plate were 1 1/2 by 2 by 1/16 in. It served to hold and align the sample in the goniometer of the x-ray diffraction apparatus.

X-RAY DIFFRACTION

The equipment used in this study was a Norelco x-ray diffraction unit operating with para-focusing geometry. K_α radiation was obtained from a copper target and nickel filter at 35 kv. and 20 ma. The collimating beam was defined by a divergence slit of 0.5° angular aperture at the x-ray port. The diffracted beam was defined by a 0.006-in. receiving slit and by a 0.5° angular aperture scatter slit. The signal of the diffracted beam was received by a Geiger-counter tube and traced by a Brown recording potentiometer.

The scale factor, multiplier, and time constant of the recording unit were set at values of 8, 0.8, and 8 sec., respectively. A goniometer scanning rate of 0.5° per min. and a chart speed of 0.25 in. per min. were used. These conditions gave a good compromise between sensitivity, stability, time, and reduction of background noise.

Before each test, the performance of the apparatus was checked by measuring the diffraction intensity from a standard brass plate set at a Bragg angle of 42.5° (80). Slight adjustments of the x-ray tube voltage were made when necessary to return the intensity to a standard value.

Diffraction pellets to be tested were placed in the holder and mounted in the goniometer. The diffraction intensity was measured from an angle of 12° to 24° 2θ. This range encompasses the three main diffraction peaks of cellulose: the 002, 101, and $10\bar{1}$. In order to eliminate possible errors due to misalignment of the pellet in the holder, the pellet was generally removed, rotated 90°, and then retested.

As described earlier, the crystallinity index was determined by measuring the intensity of the 002 diffraction peak at a Bragg angle of approximately 22.6° and the intensity of the so-called amorphous background at an angle of approximately 18°.

PREPARATION OF PULPS

DESCRIPTION OF STARTING MATERIAL

The pulp chosen for this study was a high-purity, muslin rag pulp. This provided a relatively highly crystalline starting material which contained a minimum of extraneous substances such as hemicelluloses which might be affected by treatment in the aqueous ethylamine solutions. Duplicate determinations showed the pulp to be approximately 99% alpha-cellulose (81).

DESCRIPTION OF APPARATUS

In order to minimize oxidative degradation of the pulp during treatment in the ethylamine solutions, it was necessary to design equipment which would allow not only treatment but also washing of the pulp in a nitrogen atmosphere. The apparatus used in this study is shown schematically in Fig. 1.

The cylindrical wall of the vessel was constructed of one-quarter inch Plexiglas and the cover of one-half inch Plexiglas. The bottom was one-half inch stainless steel (Type 316) with a raised lip around the edge into which the cylindrical wall fitted. Both the top and bottom were sealed with O-rings.

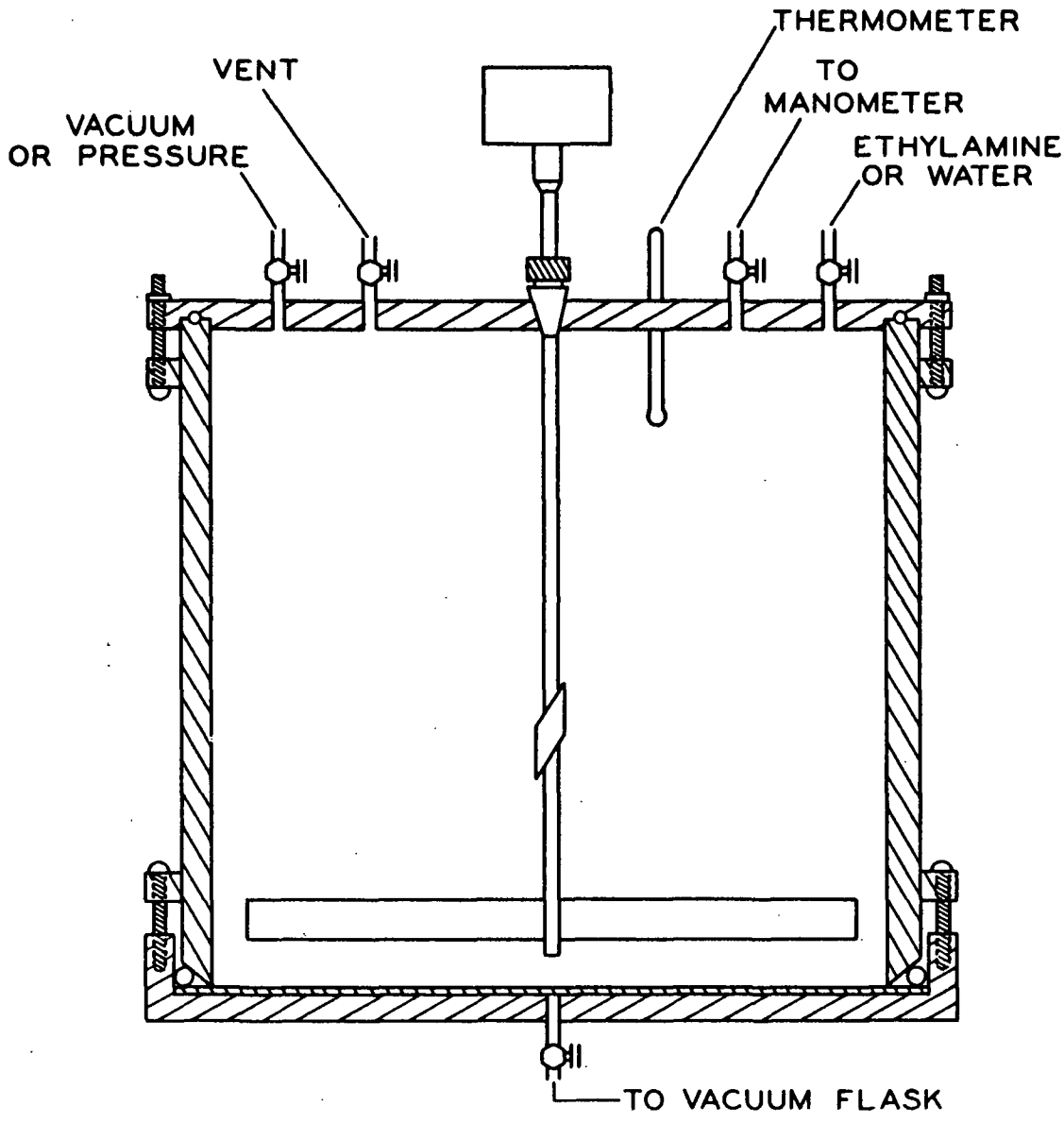


Figure 1. Large-Scale Decrystallizing Apparatus

The bottom of the vessel was fitted with a one-quarter inch stainless steel needle valve through which the ethylamine solutions and wash water could be drawn by application of vacuum. The treated pulp was retained on a 250-mesh stainless steel wire cloth which was supported by a 16-mesh stainless steel screen. The 16-mesh screen allowed relatively free movement of liquid under the 250-mesh cloth. Both pieces were held in place by the bottom O-ring.

The top of the vessel was fitted with a thermometer and with valves for the introduction of nitrogen, ethylamine, and water, and for the application of vacuum. Through another valve, the system was connected to a U-tube mercury manometer. A stirring paddle, with two blades (perpendicular to each other) reaching nearly to the wall of the vessel, was connected through a Teflon gland to a positive displacement air motor. (An electric motor was not used because of the flammability of ethylamine.)

The vessel was approximately 10.5 inches high and had an inside diameter of 11 inches, giving a working volume of approximately 15 liters.

DECRYSTALLIZATION WITH ETHYLAMINE

Determination of the Relationship Between Crystallinity Index and Concentration

Before the large-scale treatments with ethylamine could be carried out, it was necessary to determine by preliminary experiments the concentrations of aqueous ethylamine which would yield the desired degrees of decrystallization.

The sample of pulp to be treated (13 g.) was placed in a one-liter, three-necked flask fitted with a stirrer, a thermometer, and nitrogen and reactant supply lines. The flask was immersed in an ice bath and the contents were flushed with nitrogen for about fifteen minutes. The desired amount of dilution water was introduced and the vessel was flushed for another fifteen minutes. Ethylamine, previously drawn from an inverted cylinder and precooled to about -25°C., was allowed to run in by gravity. The mixture was stirred continuously for the first ten minutes and then briefly every fifteen minutes thereafter. Due to the heat of mixing of the ethylamine and water, the temperature of the slurry rose suddenly at first; it then gradually dropped to about 3°C. in about fifteen minutes. This temperature was maintained until the end of the one and one-half-hour reaction period.

At the end of this time, the mixture was poured into about one liter of cold tap water, filtered, and then washed until the wash water was neutral. In this manner, five pulp samples were treated at 2% consistency in 70, 75, 80, 85, and 94% aqueous ethylamine solutions. (A concentration of 94% was the maximum that could be attained at 2% consistency without drying the pulp before treatment.) The pulps were broken up and air dried.

Two or three diffraction disks were prepared from each pulp and at least two x-ray diffractograms were measured on each disk. From these, crystallinity indices were calculated in the manner described earlier. Typical diffractograms are shown in Fig. 2 and the tabulated indices are shown in Table I. The relationship between crystallinity index and

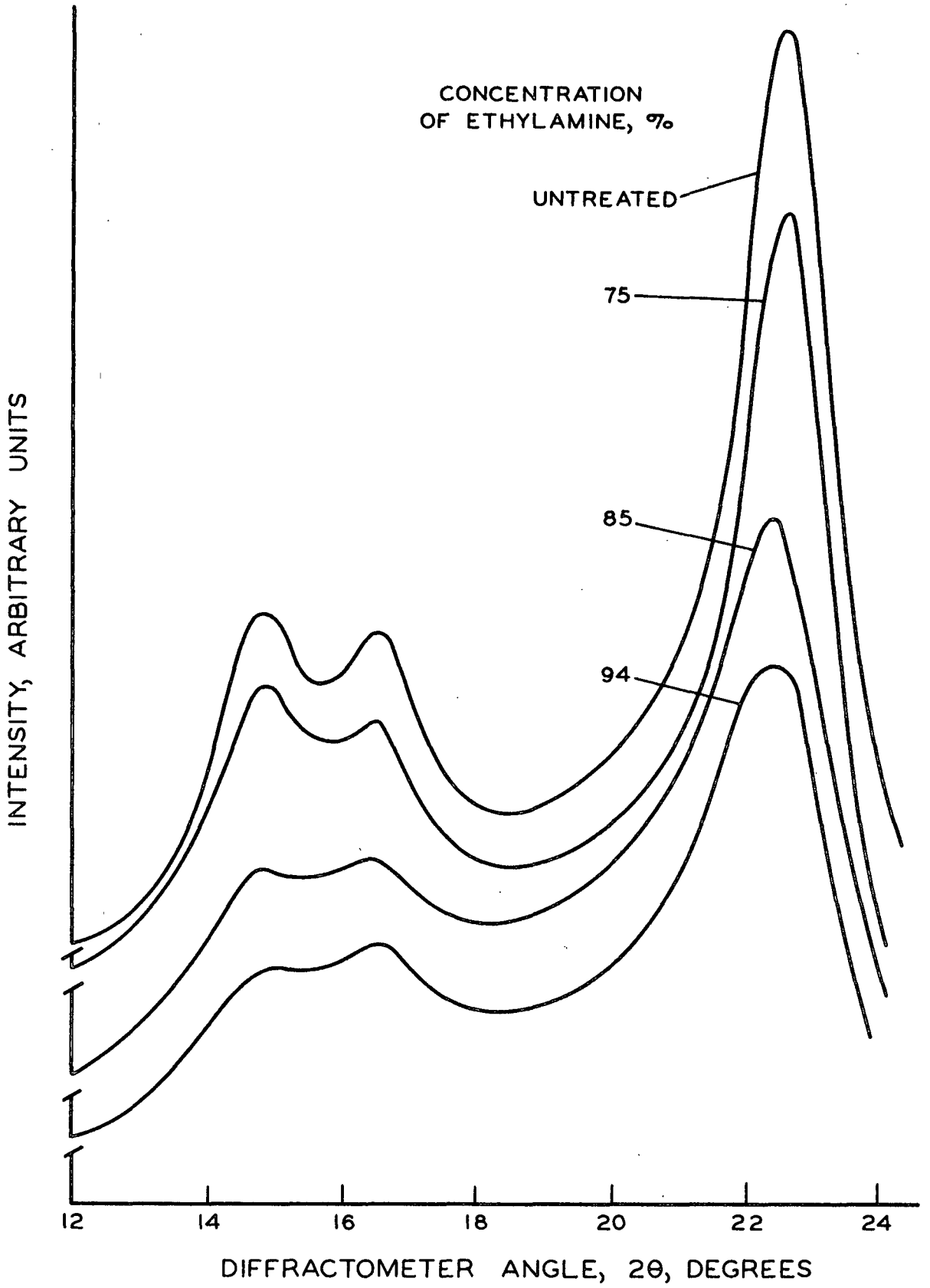


Figure 2. X-ray Diffractograms of Treated and Untreated Pulps

the concentration of ethylamine is shown in Fig. 3. As can be seen there was no apparent reduction in crystallinity below the critical concentration of approximately 71%.

TABLE I

CRYSTALLINITY INDEX AS A FUNCTION OF AQUEOUS ETHYLAMINE CONCENTRATION

		Ethylamine Concentration, %				
	Untreated	70	75	80	85	94
Cr. I	70.8	69.4	62.7	56.6	54.3	52.4
	71.0	69.4	62.7	56.8	51.8	52.8
	70.2	71.0	63.6	58.4	54.7	52.8
	68.3	71.0	62.3	56.3	52.4	50.7
				54.6	52.6	49.5
						49.5
Av.	70.1	70.2	62.8	56.5	53.2	51.5

Large-Scale Decrystallizations

With the aid of the data shown in Fig. 3 it was then possible to choose the concentrations of ethylamine necessary to yield pulps with four equally spaced levels of crystallinity index. These values were 0, 74, 79, and 94%. In addition, a concentration of 60% was chosen for preparation of a control pulp which would serve as a measure of the effects of chemical and mechanical treatment without decrystallization.

Using the apparatus described earlier, three treatments were made at each of the four concentrations of ethylamine. All reactions were run using a pulp charge of 230 g. at a consistency of 2%.

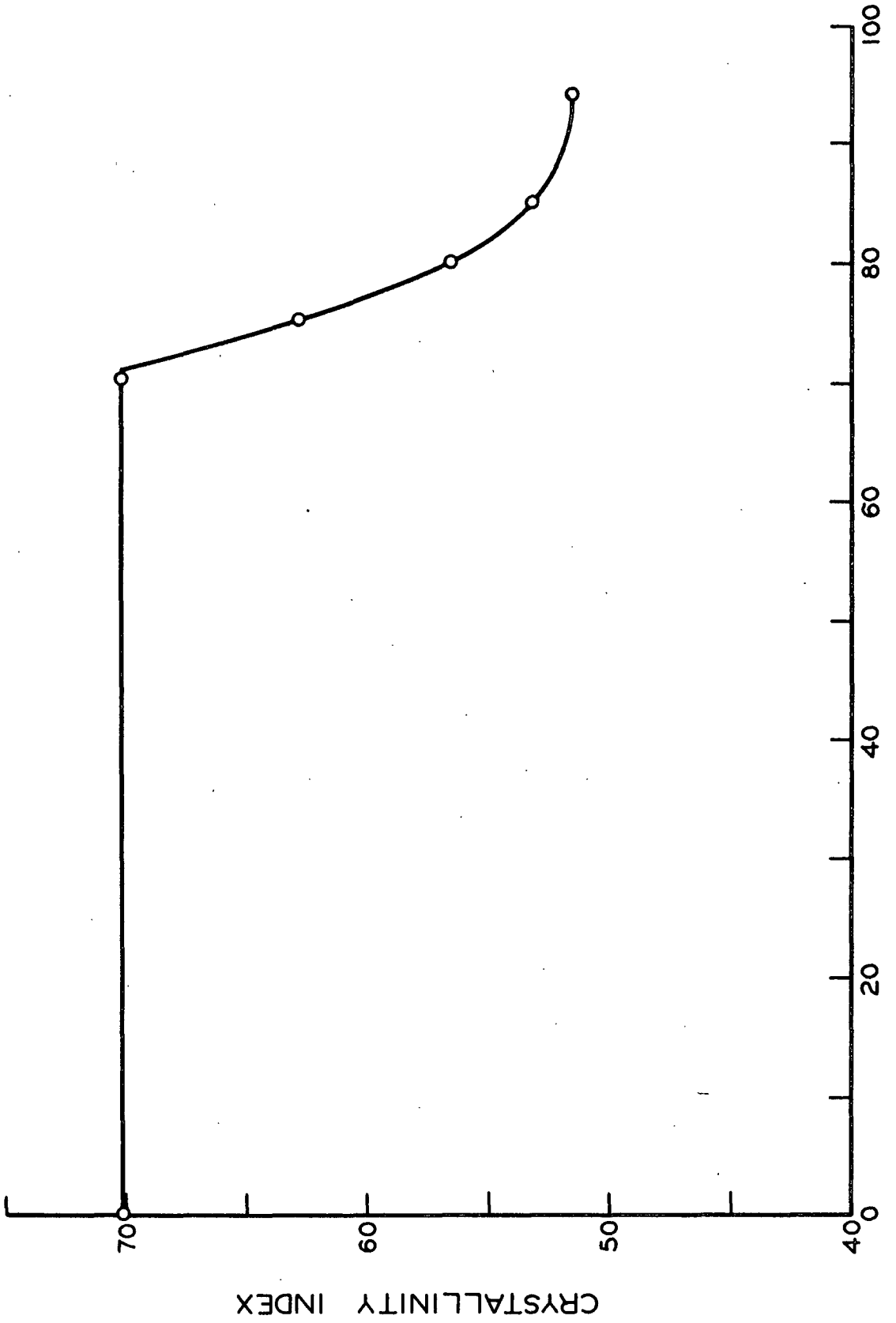


Figure 3. Crystallinity Index versus Concentration of Aqueous Ethylamine

In preliminary trials with the large-scale apparatus, it was found that if the ethylamine was first precooled to -35°C. and ice was used for dilution, the reaction mixture quickly reached a temperature near 0°C. which could easily be maintained by immersion in a salt-ice bath.

Before the reactants were added the vessel was flushed for about fifteen minutes with nitrogen. The pulp charge and dilution water (in the form of ice) were placed in the vessel and flushed with nitrogen for another fifteen minutes. The cold ethylamine was run into the vessel and the slurry was stirred frequently during the first fifteen minutes. Thereafter, the mixture was stirred for about one minute every fifteen minutes until the total reaction time of one and one-half hours had elapsed. At all times a positive nitrogen pressure of about 1 cm. Hg was maintained to insure the exclusion of air.

At the end of this time, the ethylamine was drawn off by opening the valve attached to the base of the vessel and applying suction through a two-liter vacuum flask. Care was taken to maintain a slight positive pressure within the reaction vessel at all times.

About fifteen liters of distilled water was drawn in by applying a partial vacuum to the system. The pad at the bottom of the vessel was then reslurried and allowed to stand for several minutes. This wash water was drawn out and the next portion of wash water was added. Four such washes under nitrogen were performed. The pulp was then transferred to a stainless steel pail, reslurried in distilled water,

and filtered on a table-top Buchner funnel. The resulting pad was again reslurried in the pail and dewatered. This procedure was repeated until the wash water was neutral. Generally, about eight such washes were necessary. In all cases where washing was done in the atmosphere, the mixing of the pulp in the fresh water was done by hand to minimize mechanical treatment of the pulp.

Two x-ray diffraction pellets were prepared from each run and their crystallinity indices were measured. In addition, a small sample was taken from each run and tested for intrinsic viscosity in cupri-ethylenediamine solution according to the method of Browning and Sell (82) to determine if any degradation had taken place during the treatment. These results are shown in Table II. The crystallinity indices given for each run are the averages of at least two determinations on each of two diffraction pellets. As can be seen from the viscosity results, there was no apparent reduction in degree of polymerization with treatment in the ethylamine.

The three runs at each concentration were slurried in distilled water, mixed thoroughly, and dewatered on a table-top Buchner funnel to a moisture content of about 25%. The resulting pulp mat was broken up, mixed thoroughly again, and stored in polyethylene bags at 40°F. About 2% formaldehyde (based on the ovendry pulp) was added as a preservative.

In subsequent testing of the pulps and of handsheets prepared from them, the samples were labeled according to the concentration of ethylamine in which the parent pulps had been treated. For example, the pulp

TABLE II

CRYSTALLINITY INDEX AND INTRINSIC VISCOSITY
RESULTS OF TREATED AND UNTREATED PULPS

Ethylamine Concentration, %	Run	Crystallinity Index	Intrinsic Viscosity, dl./g.
Untreated	-	70.1	12.2
60	1	69.4	12.3
	2	70.0	12.3
	3	68.8	12.4
74	3	62.4	12.4
	4	62.4	12.1
	5	63.6	12.4
79	1	54.6	12.4
	2	56.5	12.1
	3	56.8	12.3
94	1	52.0	12.4
	2	51.3	12.3
	3	52.9	12.2

treated in 74% ethylamine was labeled Pulp 74. The untreated pulp was referred to as Pulp 0. In the text of this report the properties of Pulps 0 and 60 will be compared frequently with those of Pulps 74, 79, and 94. For convenience, the former two pulps will often be referred to as crystalline pulps and the latter three as decrystallized pulps. This is not meant to imply that Pulps 0 and 60 are entirely crystalline or that Pulps 74, 79, and 94 are entirely amorphous, but is used only for ease of expression and comparison.

CHARACTERIZATION OF PULP PROPERTIES

After treatment in the various concentrations of aqueous ethylamine solutions, the resultant pulps were tested for freeness, filtration resistance, hydrodynamic specific surface and specific volume, and intrinsic fiber strength.

The Schopper-Riegler freeness of each pulp was measured according to Institute Method 414 (83). Duplicate determinations were made on each pulp and the results were corrected to a water temperature of 20°C.

Ingmanson (84) has shown the filtration resistance test to be a much better measure of pulp drainage characteristics than freeness tests. In addition, estimates may be made of pulp hydrodynamic specific surface areas and specific volumes when the tests are conducted in conjunction with pulp compressibility measurements (84-86).

The filtration tests were made under conditions of constant flow rate which allow convenience in testing and in calculation of the desired parameters (86). The average filtration resistance was determined as a function of the fluid pressure drop. The compressibilities of the pulps were determined in separate experiments by measuring the compaction of wet pulp pads as a function of mechanical stress (87). From these data, the hydrodynamic specific surface areas and the swollen specific volumes of the pulps were calculated. A derivation and discussion of the pertinent equations and calculations are not given here but may be found in the literature (84, 87-89).

As a further means of characterizing the pulp properties, tests were conducted to determine the changes in intrinsic fiber strengths which resulted from treatment in the ethylamine solutions. These tests were made utilizing the zero-span tensile-strength technique which has been shown to yield a realistic estimate of the strength of fibers composing a sheet of paper (6).

Zero-span clamping jaws were first developed by Jacobson (90) and then modified by Gunther (91) and Clark (92), among others. The jaws used in this study were developed by Wink and Van Eperen (93) who also investigated the various sheet-making variables and sheet properties which affect this test.

Samples from each of the five pulps were taken to give British-size handsheets of 50 g./sq.m. basis weight. Each sample was disintegrated in the British disintegrator for 25,000 revolutions and the sheets were formed in the British sheet mold using a 20-in. extension to the forming tube to improve formation. After couching from the wire, the sheets were pressed against stainless steel plates and dried in rings according to TAPPI Standard T 205-m. These procedures were suggested by Wink and Van Eperen (93) for obtaining optimum zero-span tensile strength results.

Two 1 by 3.5 in. strips were cut from each handsheet by cutting around a calibrated template with a fresh razor blade. Each strip was weighed for basis weight determinations.

The zero-span tensile tests were conducted by mounting the jaws in an Instron Universal Testing Instrument. (A description of this equipment is given later.) The specimens had an effective test width of 15 mm. and were loaded at a rate of 4.0 kg./cm. sec. The strengths at break were recorded automatically on a strip-chart recorder. Ten tests were conducted on the strips from each pulp and were corrected for variations in handsheet basis weights.

PREPARATION OF HANDSHEETS

DESCRIPTION OF APPARATUS

Because of the poor sheet-forming characteristics of long-fibered, high-purity cotton pulps, it was necessary to construct a sheet mold forming box which would allow high dilution of the stock before formation. An aluminum headbox, 66 in. high, was constructed to fit a 9 by 9-in. Valley sheet mold. This forming box allowed consistencies as low as 0.005% when preparing handsheets of 80 g./sq.m. basis weight.

The sheet mold was equipped with a removable septum which held a 100-mesh square-weave wire. The headbox could be clamped easily in place on top of the septum by means of two toggle clamps. Seals were made between the septum and frame and septum and forming box by means of rubber gaskets.

A sight glass was attached below the septum to allow observation of the amount of water in the mold. A valve at the base of the glass could be closed prior to the sheet forming to prevent the drawing of air into the water leg below the septum.

Tap water was supplied to the mold through the following filter system: A commercial Ful-flo filter was installed in the line followed by another Ful-flo filter wrapped with one layer of Whatman No. 1 filter paper. A special pulp filter was constructed from a six-inch piece of 8-in. diameter brass pipe which was threaded and capped at both ends. Each cap was drilled and tapped to receive a short piece of 1/4-in. pipe for hose connections. The inside of the bottom (downstream) cap

was covered with a piece of 20-mesh bronze wire cloth. This in turn was covered with two pieces of Whatman No. 1 filter paper. A pulp pad, prepared from six pieces of filter paper disintegrated in a malted milk mixer, was formed on top of the filter paper. The pad was finally covered with two more pieces of filter paper. The upper cap was screwed in place and the filter was connected to the sheet mold with thick-walled rubber pressure tubing.

A Roth centrifugal pump was installed between the second Ful-flo filter and the pulp filter to increase the flow rate. With this system, the sheet mold could be filled with water in about twenty minutes.

HANDSHEET FORMING PROCEDURES

Individual samples of pulp to give handsheets of approximately 80 g./sq.m. basis weight were weighed into small polyethylene bags. Two hundred ml. of distilled water were added to each and the bags were stored overnight in the cold room at 40°F.

When ready to prepare a sheet, a sample was placed in a jar of a British disintegrator, diluted to two liters, and disintegrated for three hundred counts (7500 revolutions).

The sheet mold was filled with water, the sight glass valve was closed, and the contents of the disintegrator jar were washed into the top of the forming box. The slurry was agitated by raising and lowering a long-handled, perforated plate ten times in about twenty seconds. The valve at the base of the mold was opened slowly and the water was allowed to drain out. When the sheet had been formed (this could be detected

most easily by listening for the sound of the first rush of air through the sheet), the valve was quickly closed and the forming box was removed from the septum. The septum and handsheet were then carried to an adjoining table.

Two standard 9 by 9-in. blotters, which had previously been sprayed lightly with a clear lacquer as a releasing agent, were soaked in distilled water. (This action prevented cockling of the blotters when they were placed next to the wet sheet.) The handsheet was covered with one wet blotter and with two fresh, dry blotters. A light, felt-covered roller was run back and forth over the sheet fifteen times with no additional pressure applied to the roll. The two upper blotters were replaced with one fresh dry blotter and the rolling action was repeated. After repeating this procedure again the sheet and wet couch blotter were lifted from the wire. (It was found when preparing sheets from Pulps 74, 79, and 94, that if more than one rolling operation were performed the sheets had a tendency to adhere to the wire because of excessive dryness. Therefore, only one such operation was performed.) The wire side of the sheet was immediately covered with the other wet, sprayed blotter.

HANDSHEET PRESSING AND DRYING PROCEDURES

In order to study the effects of interfiber as well as intrafiber properties on the viscoelastic behavior, it was necessary to find a means of widely varying the interfiber bonding without significantly affecting the intrafiber structure. The most logical manner in which to do this would of course be wet pressing. However, it was found that conventional

wet-pressing procedures did not alter sheet bonding and strength greatly. Therefore, a method of drying the handsheets under pressure, similar to that used by Sanborn (19), was chosen.

The handsheet, sandwiched between the wet couch and cover blotters, was placed between several fresh, dry blotters. When pressing at 50, 250, and 1000 p.s.i., 7, 8, and 10 fresh, dry blotters, respectively, were placed on each side of the sheet.

The handsheet and blotters were immediately transferred to a hydraulic press. The pressure was brought evenly to the desired value in thirty seconds and held until a total time of ten minutes had elapsed. The sheet was removed from the press and the blotters were carefully separated from it. The handsheet was then placed between fresh, dry blotters (five on each side) and pressed for another ten minutes. (When sheets were pressed at 250 and 1000 p.s.i. it was necessary to place dry, sprayed blotters next to the sheet during the second pressing to facilitate separation. During additional pressings they were not needed.) This procedure was repeated twice more, reducing the number of fresh, dry blotters on each side of the sheet to three and then two. At this point the handsheet was essentially dry (about 10% moisture) and was transferred immediately to a room at constant temperature and humidity (73°F. and 50% R.H.). In the case of the sheets pressed at 1000 p.s.i., only three pressings were required to dry the sheets.

PREPARATION OF TEST SPECIMENS

After conditioning for at least 24 hours, test specimens were cut from the handsheets. Eight 1 by 8-in. strips were cut from the center

of each handsheet by cutting around a steel template with a fresh razor blade. The area of a strip cut in this manner had been determined earlier by cutting and weighing strips of paper and aluminum foil of known basis weights. This area was found to be 51.1 sq. cm.

The strips were always cut and labeled in the same direction (from right to left when facing the sheet mold). Each strip was labeled as to the pulp from which it was prepared, the pressure at which it was pressed, the handsheet from which it was cut, and its position in the sheet. For example, the fourth strip from handsheet number 5, pressed at 50 p.s.i. from Pulp 74 was labeled 74-50-5-4.

Each strip was weighed on an analytical balance to a tenth of a milligram. Moisture determinations were made on each set of handsheets from a given pulp by weighing and drying the portions of the handsheets remaining after the test specimens had been cut from the center. The average ovendry basis weight of each strip could then be calculated easily.

The test specimens were divided into appropriate groups and stored in envelopes to minimize the effects of small fluctuations in room conditions.

INSTRON LOAD-ELONGATION MEASUREMENTS

The mechanical behavior of the handsheets prepared from the five pulps described above was measured on the Instron Universal Testing Instrument. Van den Akker and Hardacker (91) have studied the ability of this instrument to characterize paper properties. The Instron is

designed to apply a constant rate of straining to the specimen held between the jaws. While the upper jaw is fixed, the lower jaw is driven downward at one of several constant speeds. The upper jaw may be connected to one of several strain gages, the amplified signal of which is recorded on a variable-speed strip chart. The rate of straining may be varied from 0.02 to 50 in./min. and loads from two grams to 200 lb. may be measured.

Line-contact jaws were used in all tests. Van den Akker and Hardacker (94) and Sanborn and Diaz (95) have shown that jaws of this type are necessary for accurate measurements.

From the load-elongation curves of each test strip, values for tensile strength, ultimate elongation, and apparent elastic modulus could be calculated. The latter values were obtained by measuring the initial slopes of the curves. In general, five load-elongation tests were made on handsheets from each pulp at each wet pressure. The reproducibility between handsheets was good, with individual values generally varying from the mean by less than 5%.

All tensile and modulus values were corrected for basis weight fluctuations by expressing them in terms of load per unit cross-sectional area of dry cellulose. A cellulose density of 1.55 g./cc. was assumed for these calculations.

The tests were run on specimens conditioned at 73°F. and 50% R.H. with an initial span length of 5.00 in. and a rate of straining of 10% per minute.

TENSILE CREEP MEASUREMENTS

DESCRIPTION OF APPARATUS

The creep testing apparatus used in this study was designed by Brezinski (4, 5). A diagram of the equipment is shown in Fig. 4. The upper jaw is fixed and the position of the lower jaw is measured directly by a micrometer and an electric contact device. The jaw assemblies may be removed for specimen mounting.

To measure the position of the lower jaw, the shaft, D, is driven downward by rotating the micrometer until a needle on the lever arm contacts the jaw at A. Driving the shaft further tilts the lever arm, breaking an electrical circuit which normally runs between the lever, the block, B, and the frame. The lever arm is unbalanced so that it is normally in contact with block, B, at C, completing the circuit. A force of only 1 or 2 grams is needed to tilt the lever arm and break the circuit; this has a negligible effect on the position of the lower jaw. Proper alignment of the shaft is maintained by the pin resting against the block at E. A more complete description of this equipment is given by Brezinski (4).

In the studies by Brezinski (4) and Schulz (38), creep jaws were used that held the specimen by mechanical clamping. These clamps were relatively heavy (about 300 g.) which presented problems in making creep recovery measurements. Recovery measurements were made by one of two techniques. In the first, the specimen was allowed to support the weight of the lower clamp during the recovery period. This of course made

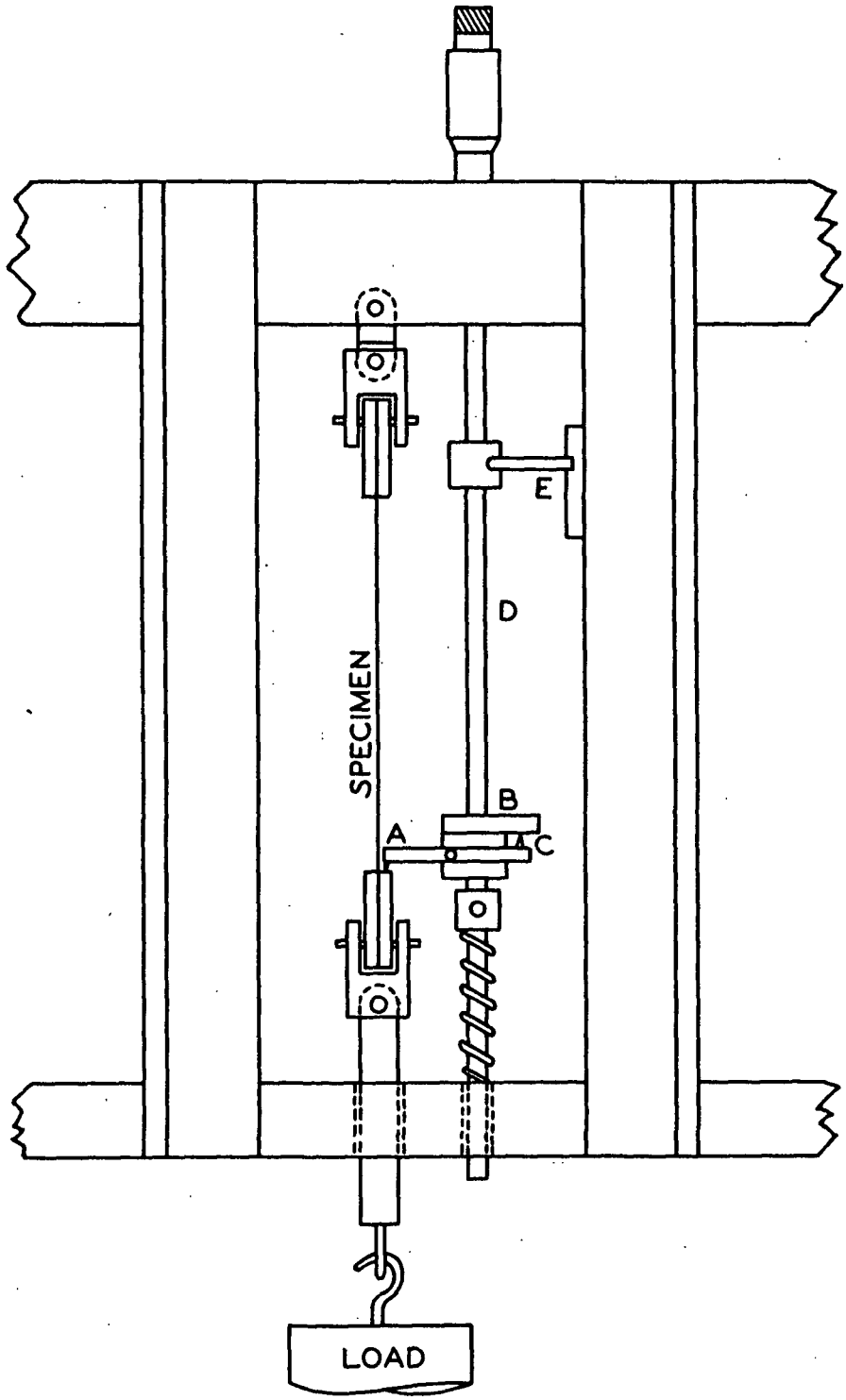


Figure 4. Tensile Creep Testing Apparatus

interpretation of the results difficult, since the creep caused by the weight of the clamp was superimposed on the recovery deformation. In general, the clamp weight was about 10 to 15% of the average creep load.

In the second technique, the lower clamp was supported by a yoke during the recovery period so that no load was allowed to bear upon the specimen. When a measurement was to be taken, the clamp was released from the yoke and allowed to draw the specimen taut. After reading the micrometer, the clamp was again supported by the yoke. When calculating the deformation, it was then necessary to correct for the specimen extension (assumed to be elastic) caused by the weight of the clamp. This correction was usually about 3.0 mils. This procedure of course necessitated considerable handling of the specimen with resultant errors due to possible misalignment of the lower clamp with each reading. In addition, the measured recovery deformations were a combination of the true recovery relationship and the cyclic behavior caused by application and removal of the clamp weight.

In view of these difficulties, it was felt that relatively light-weight creep jaws should be designed which, if allowed to remain on the specimen during the recovery period, would impose a negligible error on the measured recovery. They would also eliminate the problem of excessive handling of the samples.

However, with such lightweight jaws, some means other than mechanical clamping had to be used for holding the specimen, since such clamping requires a rigid and relatively massive system. Consequently, a set of magnesium jaws was designed and constructed in which the specimen was

held by cementing with an epoxy resin. A sketch of the jaws is shown in Fig. 5.

The jaw faces, F, to which the specimen is cemented, are aligned by steel pins, P, so that the leading edges are square. The faces and support shaft, S, are held in the yoke, Y, by steel support pins which form a universal joint. In the lower jaw assembly, all parts except the alignment pins and support pins are constructed of magnesium. In the upper jaw assembly, the support shaft is constructed of steel. The lower end of the bottom support shaft contains an eyelet from which the load may be hung. The upper support shaft is held in the frame by a steel pin.

The jaw faces are 1-1/4 in. wide, 1-3/4 in. long, and 3/16 in. thick. The entire lower assembly, including the support shaft, weighs 78 g. (This is about 2% of the average load used in this study.)

An epoxy resin was chosen for cementing the specimens in the creep jaws for the following reasons (96):

(1) The adhesive has a shear strength substantially higher than that of the specimen.

(2) The adhesive cures at room temperature.

(3) No solvent is released during the cure.

(4) No stresses are set up in the specimen during the cure.

The resin used is manufactured by the Shell Chemical Company under the trade name of Shell Epon Adhesive.

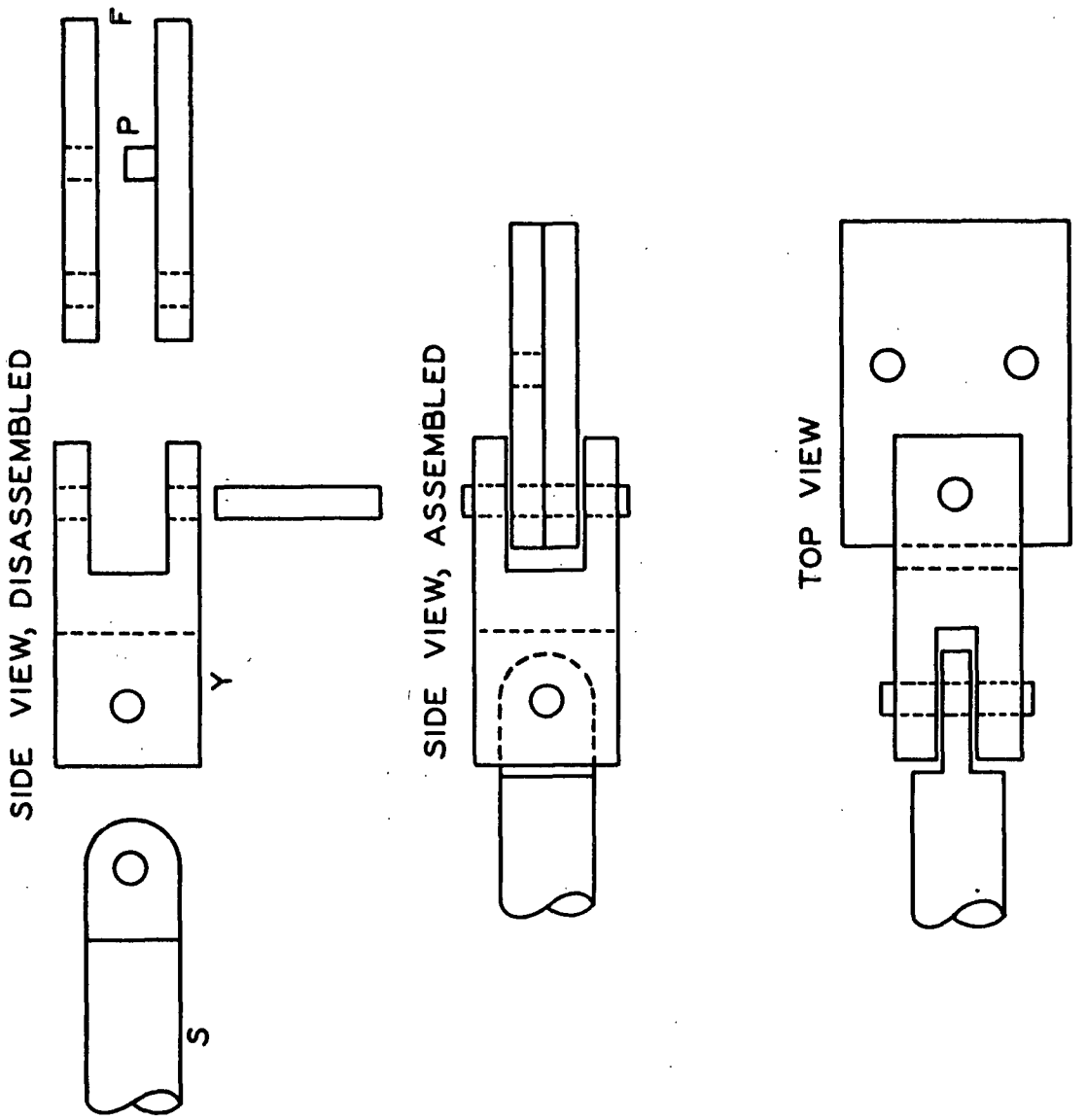


Figure 5. Lightweight Creep Testing Jaws

DESCRIPTION OF CREEP-TESTING TECHNIQUES

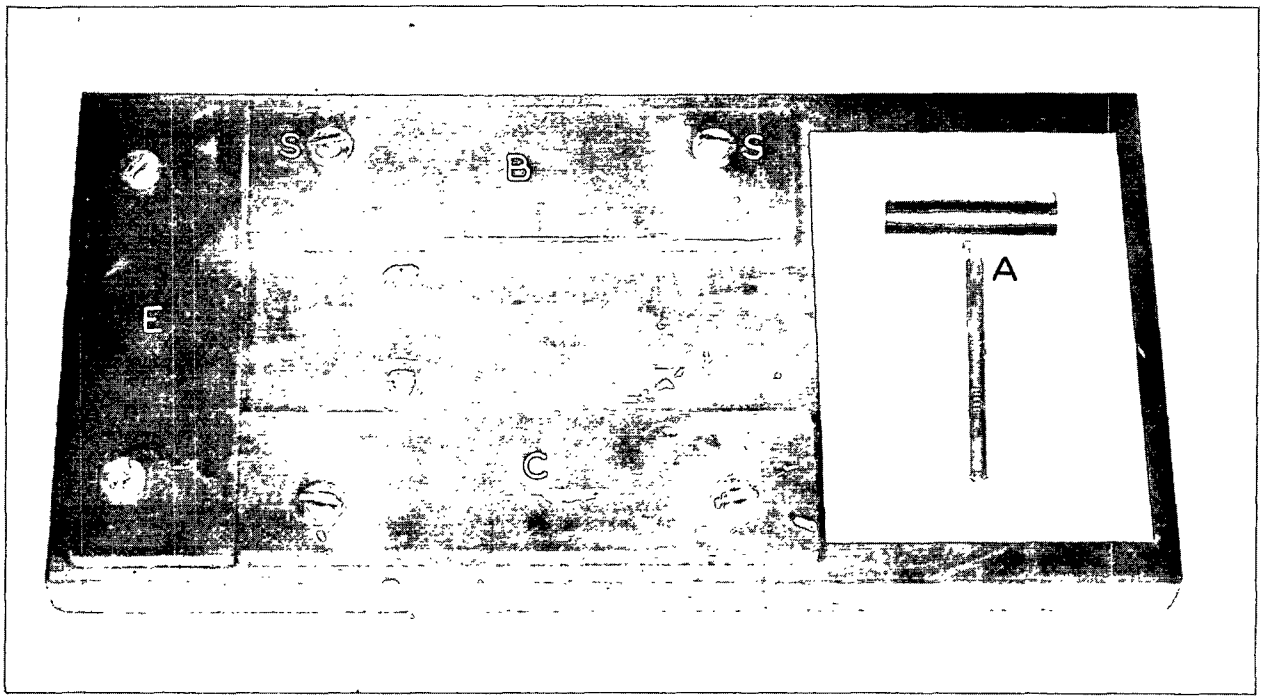
Specimen Mounting and Alignment

Figure 6 shows the jig and applicator constructed for application of a uniform and reproducible layer of resin to the creep jaws. The two steel bars, C and E, are fastened firmly to the brass base plate. The upper bar, B, may be moved slightly by loosening the two screws, S, set in oversized holes. The two jaw faces of one set of creep jaws are clamped in position, as shown in the lower photograph, by pressing bar B against the sides of the faces and tightening the screws, S. The bars, B and C, are of the same thickness as the jaw faces.

The applicator, A, is a piece of 3/8-in. diameter brass rod, 1-1/8 in. long. The center section of the rod, one inch wide, has been machined to a diameter two thousandths of an inch less than the ends. A small handle is attached for ease of handling.

When applying the resin, the two jaw faces to be coated were clamped in position as described above. An adequate quantity of the resin was placed at B. The applicator was then drawn slowly across the jaw faces from B to C, thus applying to each face, simultaneously, a uniform coating of resin, one-half inch wide and approximately 0.002 in. thick.

When two sets of jaw faces had been treated in this manner, one face of each set (that containing the alignment pins) was clamped in the specimen mounting jig as shown in Fig. 7a. This jig had been constructed



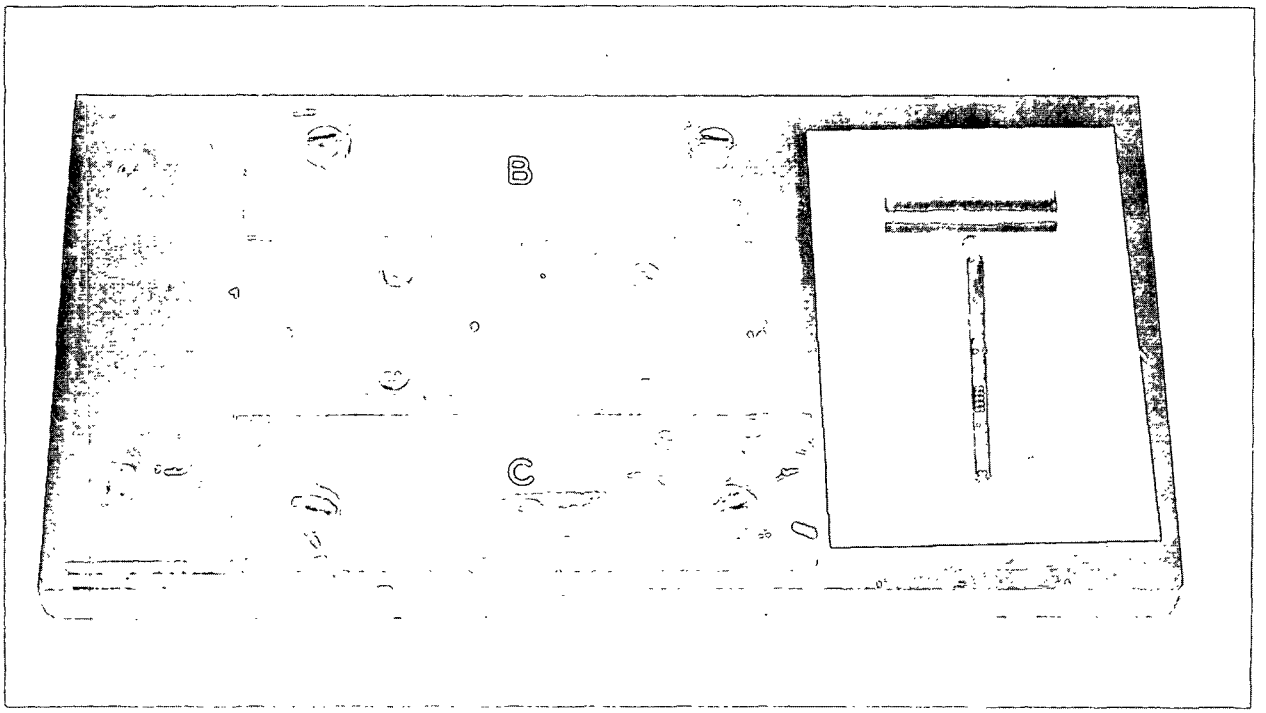


Figure 6. Jig for Application of Epoxy Resin

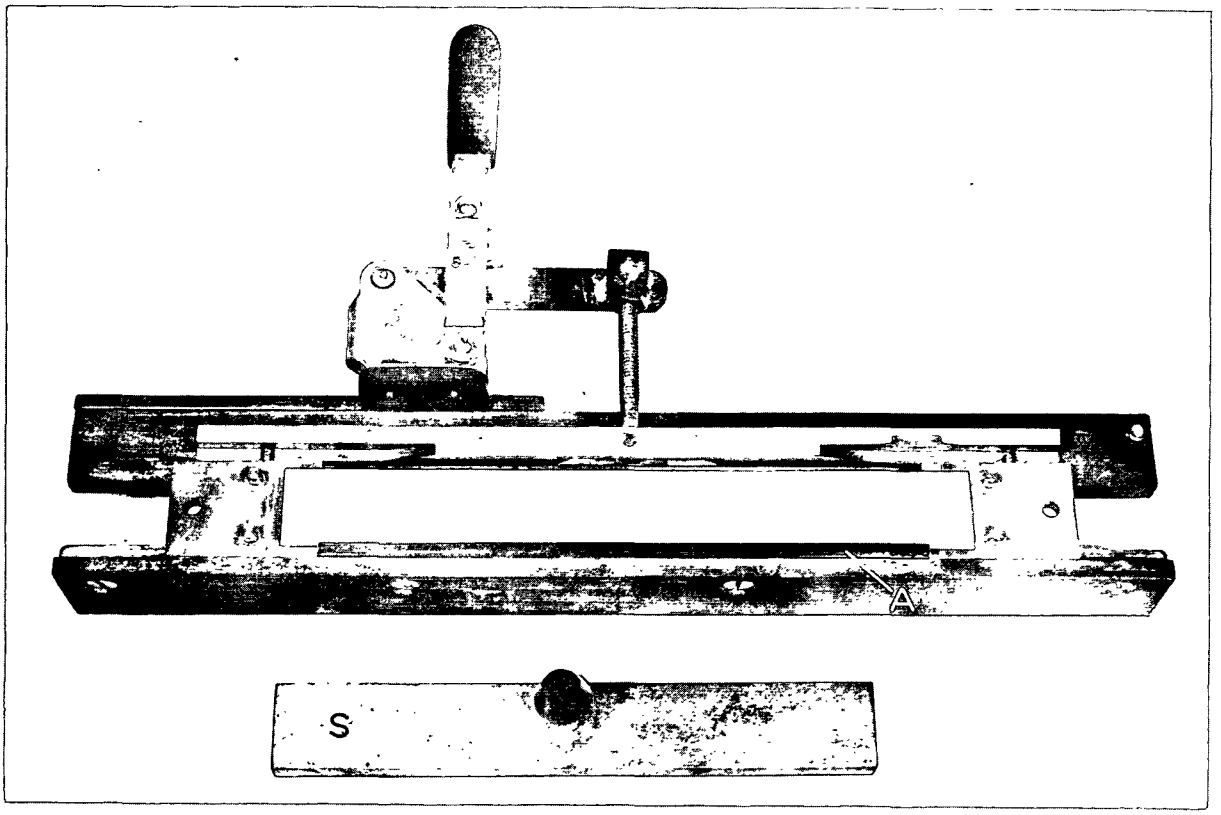


Figure 7a. Alignment Jig with Specimen and One-half of Each Jaw in Place

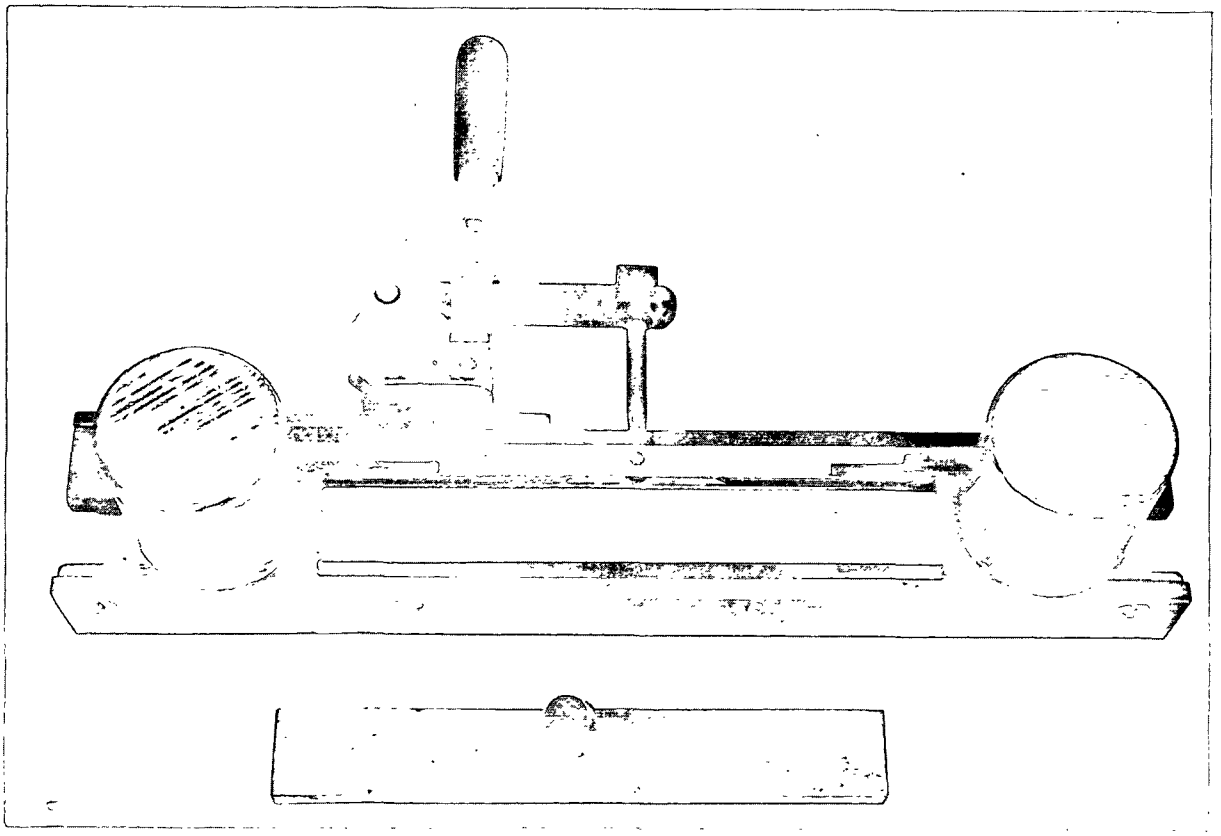


Figure 7b. Alignment Jig with Jaws, Specimen, and Weights in Place

to allow proper and reproducible alignment of the creep specimens in the jaws. It also maintained a constant initial span length of 7.00 in. between the jaws.

The specimen, 1 by 8 in., was carefully placed on the platform between the two jaw faces with one edge pressed against the spacing bar, A, which aligned the strip in the centers of the jaws. (All specimens were handled with forceps to prevent contamination.) One-half inch at each end of the strip was then placed in contact with the resin on the jaw faces. A steel bar, S, one inch wide and 6-3/4 in. long was placed on top of the specimen to insure flatness, and then the two upper faces of the jaw sets were put in place. After removing the steel bar, S, two 1-kg. weights were placed on top of the jaws. (See Fig. 7b.) The entire assembly was allowed to remain in this state for at least four hours, at which time the resin had essentially cured. The jaws and specimen could then be removed to allow mounting of another specimen. In general, however, specimens were not tested until the following day.

When tests on a specimen had been completed, the two sets of jaw faces were separated from their yokes and the center of the test strip was cut away. The jaw faces were then soaked in absolute ethanol for about two hours. At this time, the two halves of the jaw could be separated and the resin and specimen residue stripped from the metal. (Absolute ethanol must be used since the magnesium jaws will react with any water present.) When not in use, the jaw parts were stored in a desiccator over calcium chloride.

Creep and Recovery Testing

When ready for a test, the specimen and attached jaw assemblies were mounted in the creep testing apparatus as shown in Fig. 4. As soon as the position of the lower jaw had been determined, the desired creep load was applied. Care was taken to load the specimen as quickly as possible without imparting a sudden impulse to it. Time of application was estimated at about 0.5 sec. Time was measured from the instant of application of the load and the first deformation-time reading was taken as quickly as possible. In general, this could be done in about 10 seconds.

Because even the relatively low weight of the lower jaw assembly had some effect upon the test specimens, the initial readings had to be corrected for slight deformations. These deformations were assumed to be elastic and the corrections for the different pulps at different wet pressures of sheet preparation were calculated using measured moduli of elasticity. The corrections ranged from 0.4 to 0.7 mils.

From the basis weights and widths of the test strips, the apparent cross-sectional area of solid cellulosic material in each specimen could be calculated. In making these calculations, a cellulose density of 1.55 g./cc. was assumed. The cross-sectional area in each case was used to calculate the apparent initial stress applied to the specimen. This stress is defined as the total applied load, in kilograms, divided by the cross-sectional area of ovendry cellulose, in square millimeters. The apparent initial stress in all creep tests included the weight of the lower jaw assembly.

All recovery tests were made with the specimen supporting the weight of the lower jaw assembly. In a manner analogous to that of the creep tests, deformations were measured from the instant of removal of the load. Although the applied recovery stresses (the removal of a load is thought of as the application of a negative load) were less than the initial creep stresses by an amount equal to the stress of the lower jaw assembly, all recovery data are labeled with the apparent initial stress of the corresponding creep tests. This avoids confusion and allows more rapid reference between corresponding creep and recovery tests. In all cases, however, the initial recovery stress was 0.06 kg./sq.mm. less than the initial creep stress.

All deformations were calculated as a percentage of the specimen length existing immediately prior to the application or removal of load. In the first-creep tests, this initial length was 7.00 in. In subsequent tests, however, the initial length was corrected for the deformation which took place in all previous tests.

Although the deformations occurring during the recovery tests are opposite in sign to those during creep tests, all were regarded as positive and are thus reported.

All creep and recovery tests were run at conditions of 73°F. and 50% R.H.

EXPERIMENTAL RESULTS AND DISCUSSION

THE EFFECTS OF ETHYLAMINE TREATMENT ON PULP PROPERTIES

Table III lists the results of the tests for freeness, filtration resistance, swollen specific volume, and hydrodynamic specific surface area for the five pulps studied. Each value given is the average of two determinations.

TABLE III

RESULTS OF FREENESS AND FILTRATION RESISTANCE TESTS

Pulp	0	60	74	79	94
Crystallinity index	70.1	69.4	62.8	56.0	52.1
Schopper-Riegler freeness, ml.	600	600	700	710	730
Filtration resistance, 10^{-8} cm./g.[a]	5.69	5.26	3.36	3.15	2.88
Specific volume, cc./g.	2.40	2.38	3.02	3.08	3.00
Specific surface, sq.m./g.	4.10	3.92	3.52	3.35	3.24

[a]Measured at a fluid pressure drop of 10 cm. water.

In Fig. 8, the filtration resistances and the corresponding specific surface areas of the five pulps are shown as a function of the pulp crystallinity index. It should be noted here that these relationships, especially that of the filtration resistance, are similar in shape to those found for most of the properties of both the pulps and the hand-sheets when studied as a function of crystallinity index: that is,

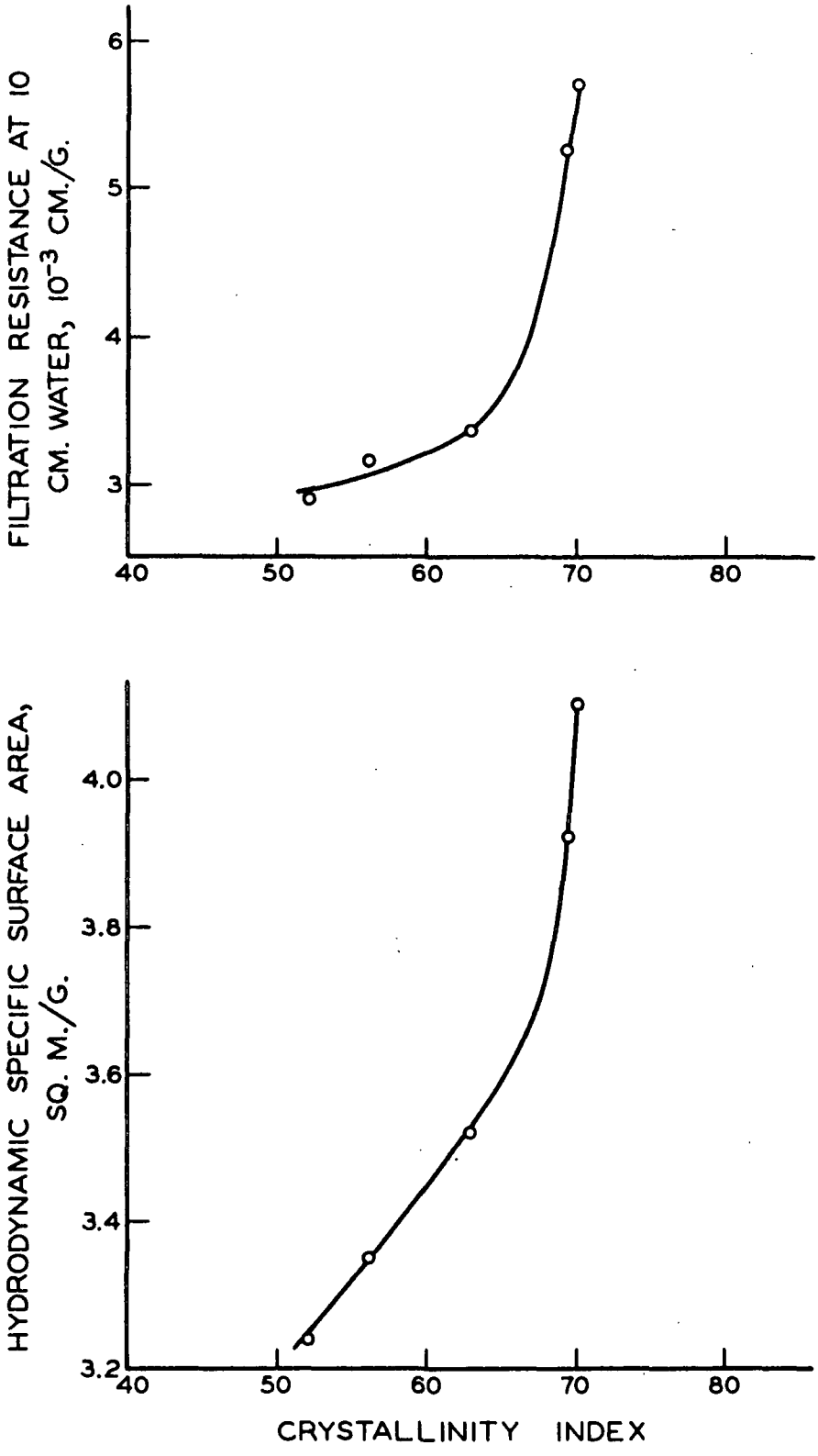


Figure 8. Filtration Resistance and Specific Surface versus Crystallinity Index

there was an abrupt change in the properties observed upon going from the crystalline pulps (Pulps 0 and 60) to the decrystallized pulps (Pulps 74, 79, and 94), with relatively small differences existing between the properties of the three decrystallized pulps.

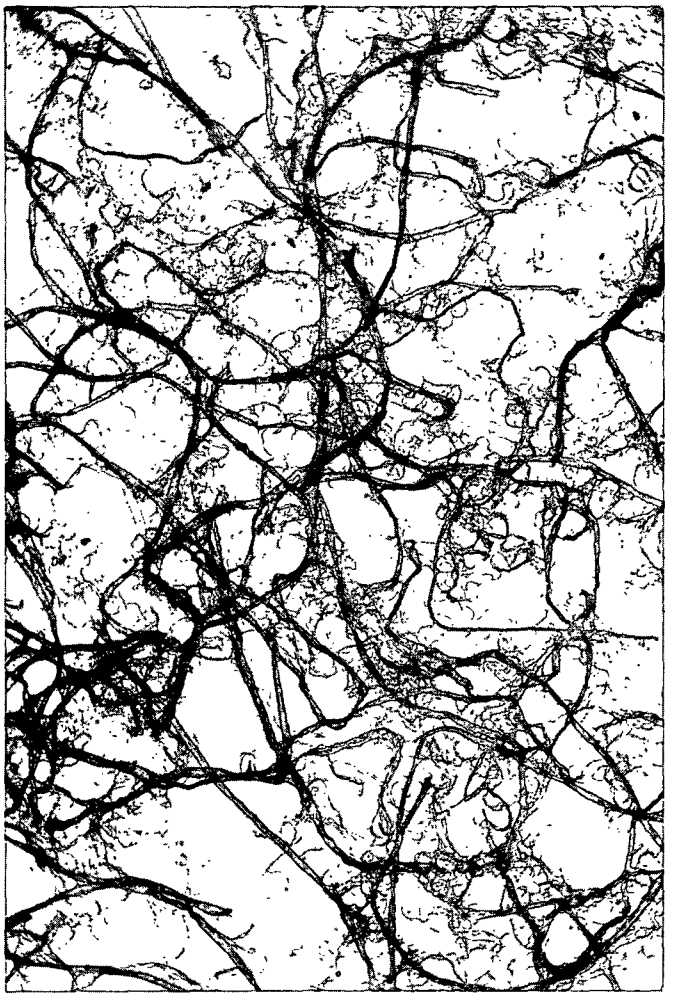
This is demonstrated further in Table IV where the zero-span tensile strengths of handsheets prepared from the five pulps are compared to the corresponding crystallinity indices. Again there is an abrupt change upon going from the crystalline pulps to the decrystallized pulps. Thus, it may be concluded that these changes in properties are due not merely to changes in fiber crystallinity but are brought about in part by the decrystallizing treatment per se, that is, treatment in a concentration of aqueous ethylamine greater than the critical 71% discussed earlier.

TABLE IV

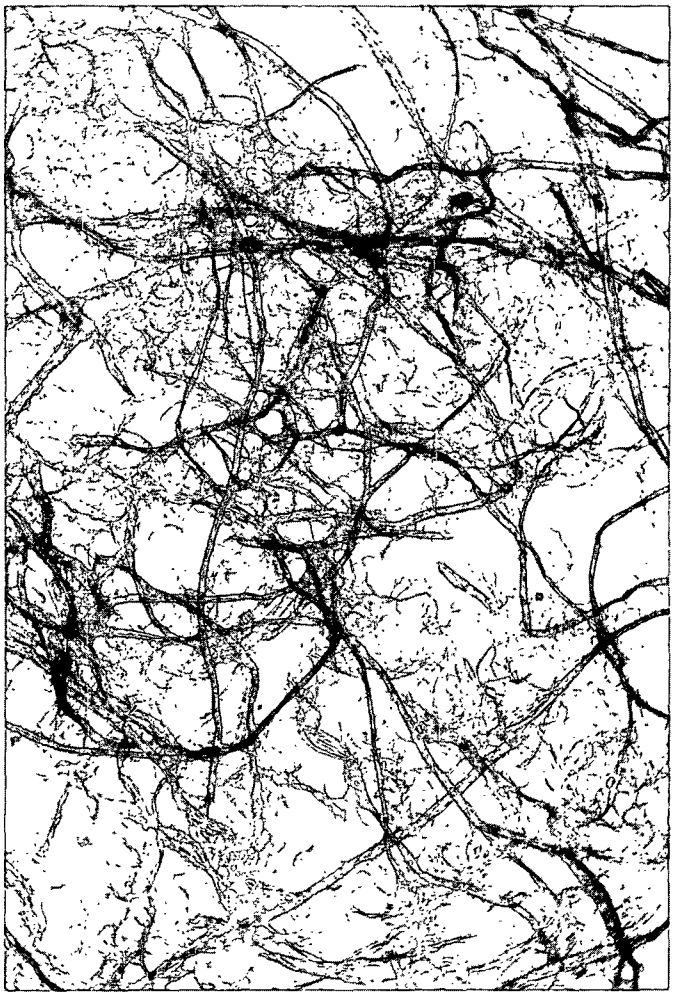
ZERO-SPAN TENSILE STRENGTHS AS A FUNCTION OF CRYSTALLINITY INDEX

Pulp	0	60	74	79	94
Crystallinity index	70.1	69.4	62.8	56.0	52.1
Zero-span tensile strength, kg./sq.mm.	24.8	24.7	21.0	20.9	20.2

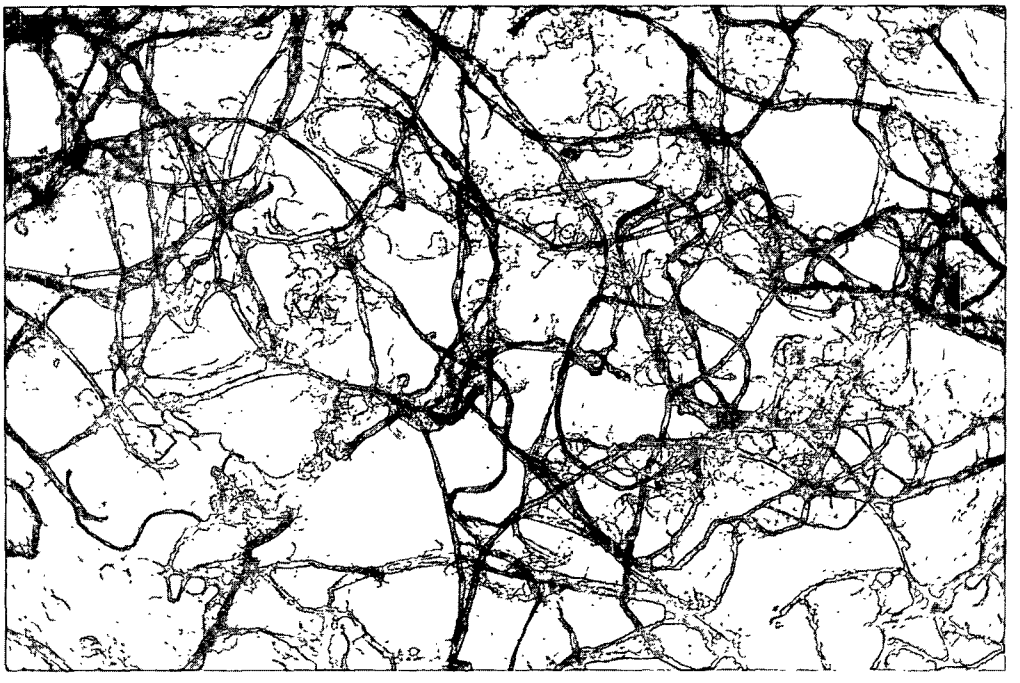
The more continuous decrease in specific surface area shows that a certain amount of fines material was lost in the ethylamine treatment and that this amount was dependent upon the concentration of the solution. (This may be seen in the photomicrographs shown in Fig. 9.) Apparently, this material was lost through the wire cloth at the bottom of the



Pulp 74



Pulp 0



Pulp 94

Figure 9. Photomicrographs of Pulps 0, 74, and 94
Magnification 60X

reaction vessel during the treatment and washing procedures. Perhaps, as the concentration of amine was increased, fines material attached to the fibers was more easily loosened by agitation and washed away.

The abrupt change in the swollen specific volume is of considerable interest and importance. It can be seen from Table III that this increase with swelling treatment is of the order of 25%. (The variation in volumes of the three decrystallized pulps is not statistically significant at the 90% confidence level.) In contrast to this, Segal, et al. (75) have measured the pycnometric density of dry cotton after treatment in anhydrous ethylamine and have found it to differ from that of the untreated cotton by less than 0.2%. This small decrease was attributed to a decrease in the amount of crystalline material in the cellulose. Thus, it is suggested that the increase in swollen specific volumes found here may be an effect of the treatment in decrystallizing concentrations of ethylamine, and is not due merely to differences in crystallinity. Although the cause of this phenomenon cannot be defined rigorously at this time, it is suggested that it may be due to a weakening of the bonding between the grosser units of the fibers--that is, the fibrils and microfibrils--which allows a considerably greater swelling of the fibers upon immersion in water. Evidently, this weakening is not a function of concentration once the concentration is greater than 71%.

The abrupt decrease in zero-span tensile strength shown in Table IV is of considerable magnitude--of the order of 15%. This is difficult to explain in view of the work done on textile yarns (59, 64, 65), if it is assumed that the zero-span technique closely reflects the true

strengths of the fibers. While Tripp, et al. (65) have reported a 6% decrease in tensile strength of cotton yarn with treatment in anhydrous ethylamine, Susich (64) has reported an increase of about 4% with similar treatment. Thus, it would appear that the treatment does not necessarily reduce the fiber strength.

It must be assumed at the present time that the reduction in strength is not due to either a reduction in cellulose degree of polymerization or to a reorientation of crystallites with treatment. The intrinsic viscosity data given earlier in Table II negate the first explanation. In regard to the second, Segal, Nelson, and Conrad (61) found that there was no significant change in crystallite orientation, as measured by an x-ray diffraction technique, with treatment in ethylamine.

It is suggested that the decrease in zero-span tensile strength is the result of at least two factors: First, the phenomenon suggested earlier in regard to the abrupt increase in specific volumes of the pulps, namely, the possible weakening of the bonding within the macrostructure of the fibers, may result in an over-all fiber strength reduction which is not highly dependent upon ethylamine concentrations greater than 71%. Second, the drop in zero-span tensile may partly reflect the decrease in bonding of the test sheets brought about by the loss of fines in the pulp. Wink and Van Eperen (93) have shown the zero-span test to be dependent upon handsheet fiber-to-fiber bonding, and although the effect is relatively small, the manner of preparing optimum zero-span test sheets from the treated and untreated pulps may differ enough to have a significant effect on the final results.

INSTRON LOAD-ELONGATION RESULTS

In the following discussion of the mechanical and viscoelastic properties of handsheets prepared from the various pulps, data will be presented for all five pulps only in certain instances. Since the differences between Pulps 0 and 60 and between Pulps 74, 79, and 94 have in almost all cases been found to be relatively small, most of the analyses and conclusions will be drawn from the data of Pulps 0, 74, and 94. This procedure in no way affects the results or conclusions which would have been arrived at had data for all pulps been presented. In some cases, however, all five pulps will be included in the discussion for the sake of completeness.

In Fig. 10-12 are presented typical load-elongation curves for Pulps 0, 74, and 94 at handsheet wet pressures of 50, 250, and 1000 p.s.i., respectively. These particular curves were chosen to best represent the observed average values of ultimate strength, elongation, and apparent elastic modulus. The ordinate of each figure, the apparent stress in kg./sq.mm., was calculated for each curve in the same manner as the "apparent initial stress" used in the creep tests. The "sq.mm." term refers to the apparent cross-sectional area of the test specimen and was calculated by assuming a dry cellulose density of 1.55 g./cc. In this way, results were corrected for variations in basis weight between samples.

In Table V are presented the average values of tensile strength and apparent elastic modulus for each of the five pulps and for each of the three wet pressures. The corresponding crystallinity indices are given

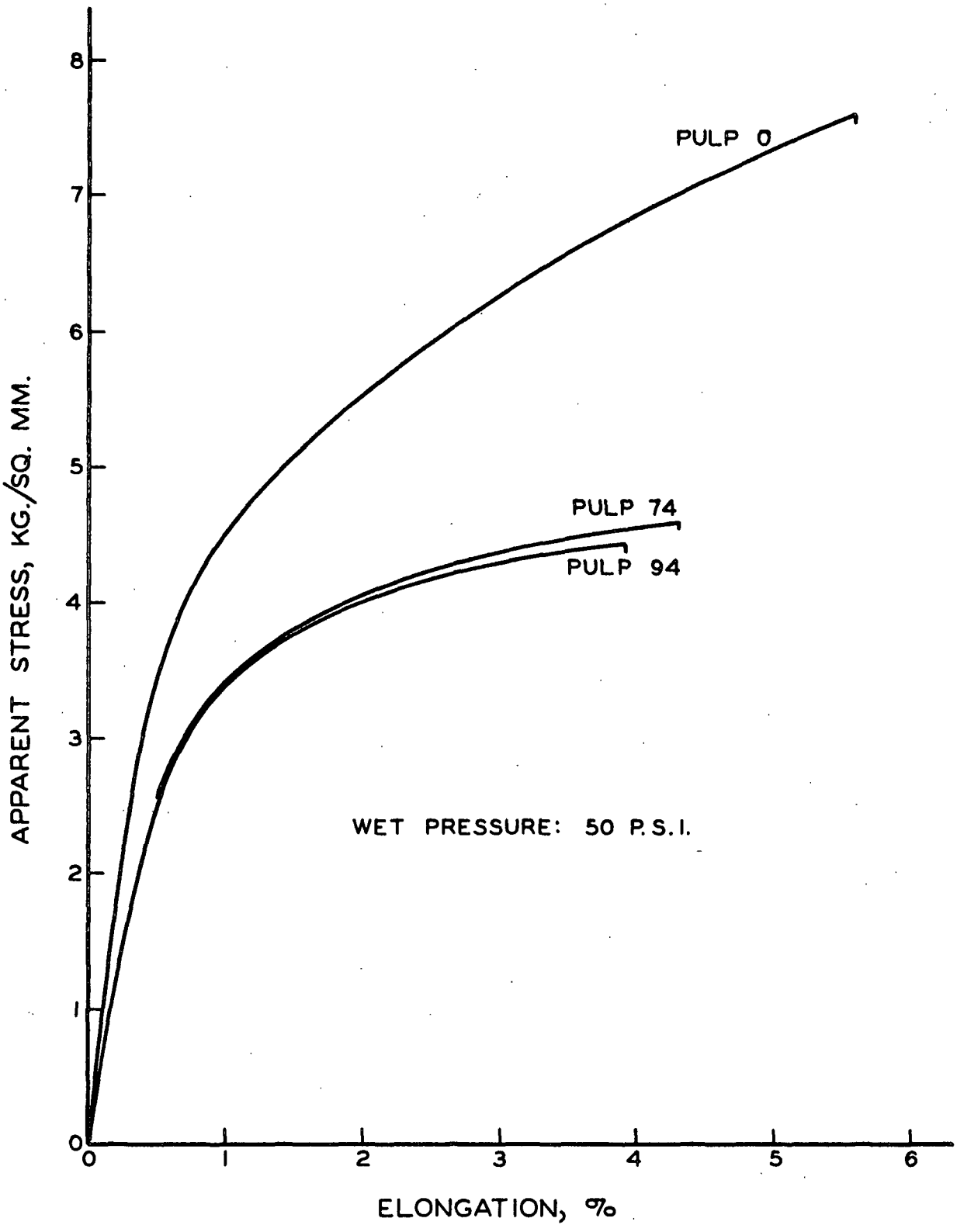


Figure 10. Load-Elongation Curves of Pulps 0, 74, and 94 at 50 p.s.i. Wet Pressure

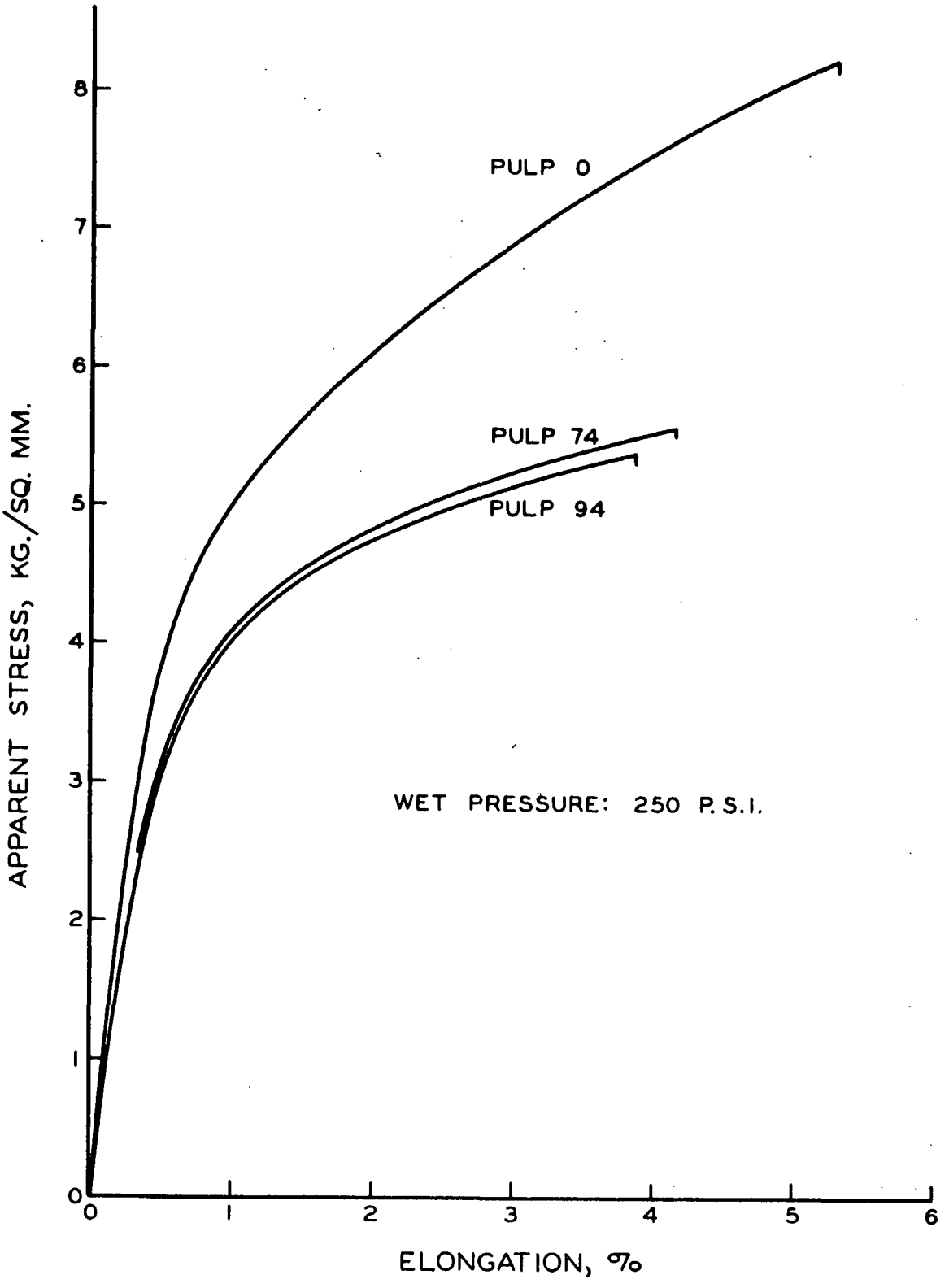


Figure 11. Load-Elongation Curves of Pulps 0, 74, and 94 at 250 p.s.i. Wet Pressure

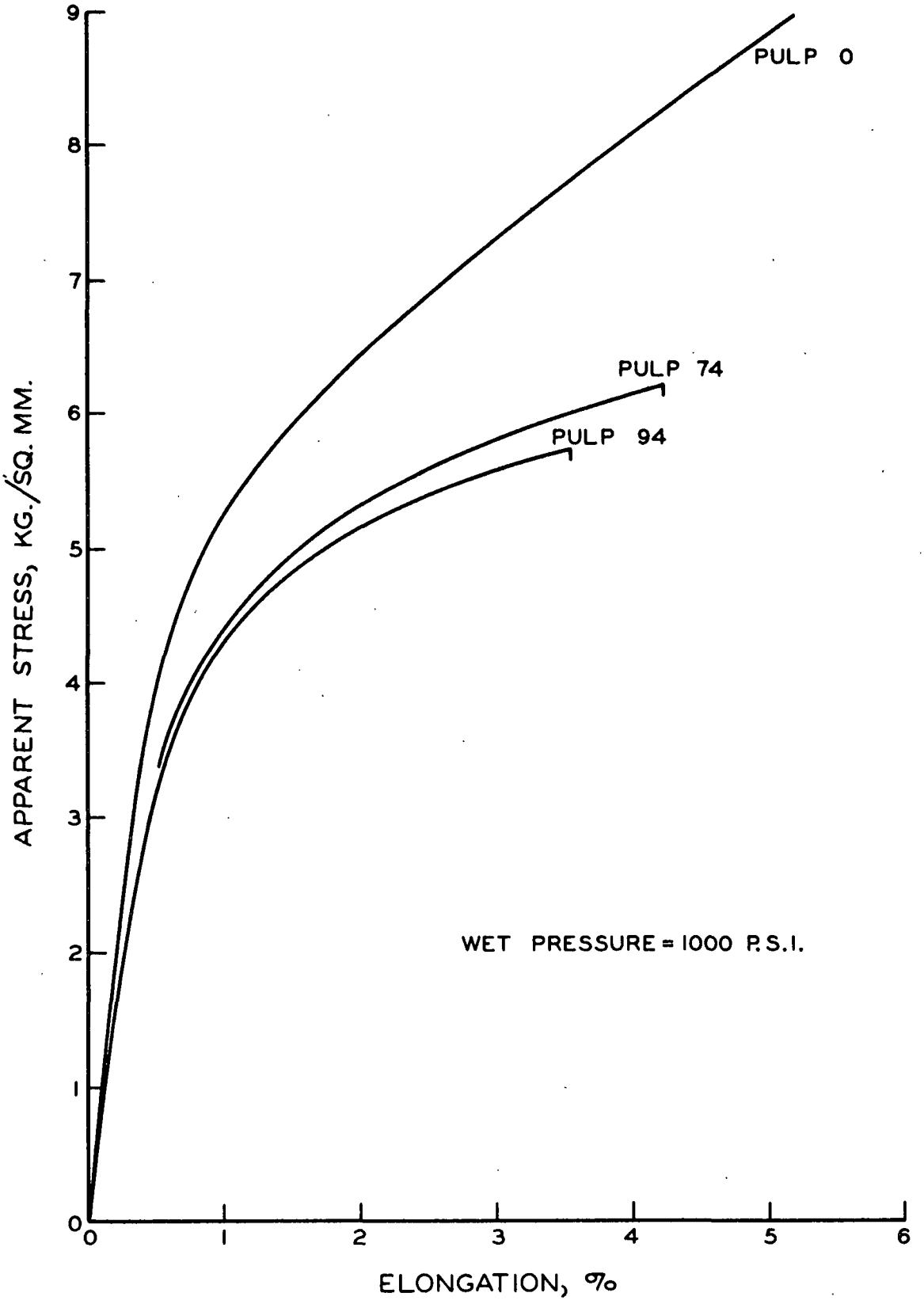


Figure 12. Load-Elongation Curves of Pulps 0, 74, and 94 at 1000 p.s.i. Wet Pressure

for comparative purposes. Each value given is the average of five tests. As mentioned earlier, the apparent elastic moduli were determined by measuring the initial slopes of the load-elongation curves. The results listed in Table V are shown graphically in Fig. 13 with handsheet wet pressure as the parameter.

TABLE V

TENSILE STRENGTHS AND APPARENT ELASTIC MODULI OF TEST HANDSHEETS

Pulp	0	60	74	79	94
Crystallinity index	70.1	69.4	62.8	56.0	52.1
Equil. solids content, %	93.7	93.5	93.0	92.5	92.5
			Tensile Strength, kg./sq.mm.		
Wet Pressure, p.s.i.					
50	7.3	7.4	4.6	4.4	4.4
250	8.2	8.4	5.6	5.4	5.4
1000	9.0	9.0	6.2	6.0	5.8
			Apparent Elastic Modulus, kg./sq.mm.		
50	880	856	645	623	610
250	980	965	805	782	770
1000	1010	991	815	804	787

The most obvious characteristic of the data presented in Fig. 10-13 is again the abrupt change upon going from Pulps 0 and 60 to Pulps 74, 79, and 94. Whereas the difference between the tensile strengths of Pulps 0 and 74 is about 37% at 50 p.s.i., that between Pulps 74 and 94 is only about 4%. (The ultimate elongation is seen, in Fig. 10-12, to behave in a similar manner.) Thus, it must again be concluded that the main decrease in tensile strength is due primarily to some effects of the decrystallizing treatment other than changes in the degree of crystallinity.

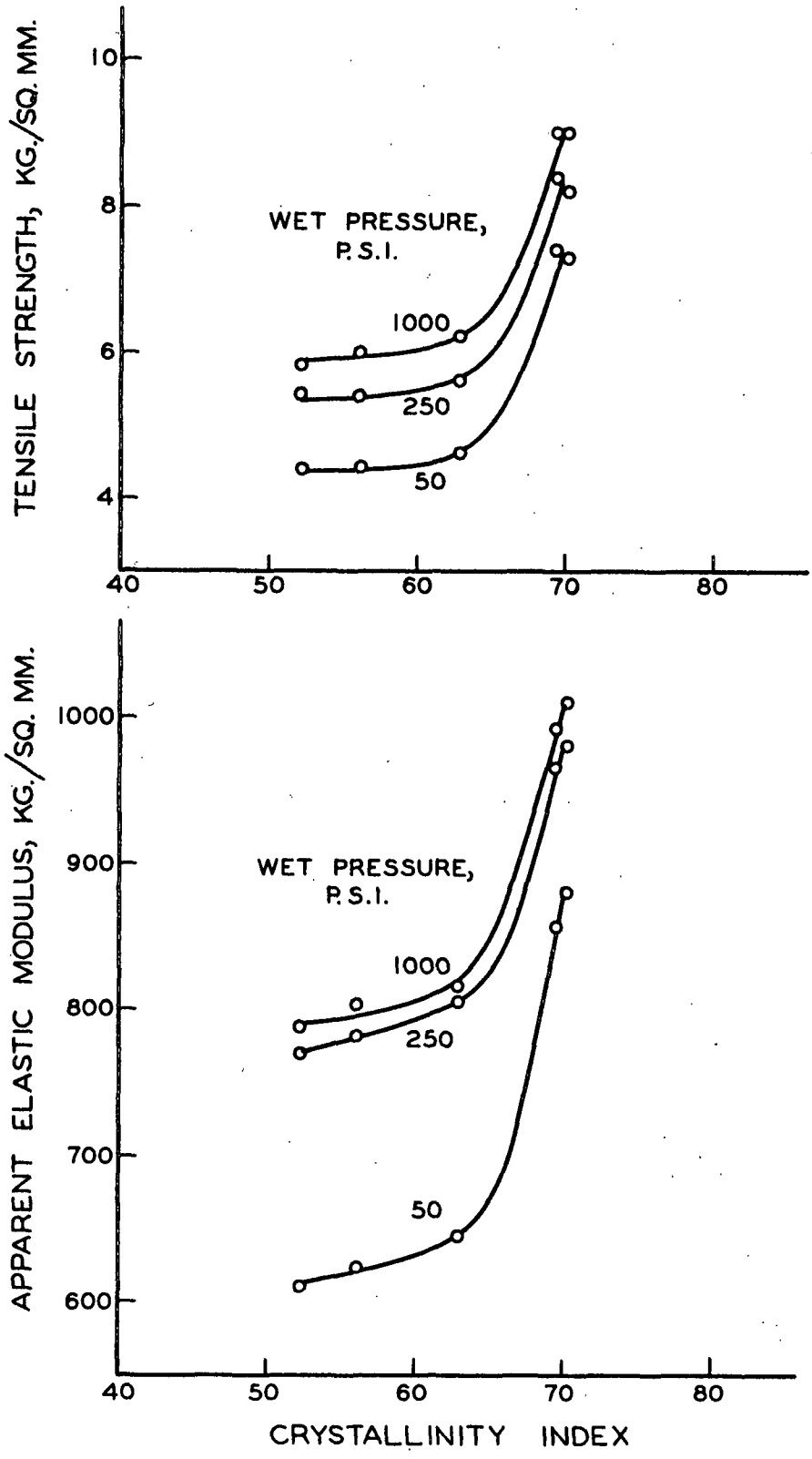


Figure 13. Tensile Strength and Apparent Elastic Modulus versus Crystallinity Index

Since the tensile strength is dependent upon the extent of fiber-to-fiber bonding within the sheets, it is suggested that the marked change in this property with treatment is caused in part by the substantial decrease in surface areas of the decrystallized pulps. Of course, a real difference in the fiber strengths of the decrystallized and crystalline pulps could also play a part, since Van den Akker, et al. (6) have demonstrated the importance of fiber strength to sheet strengths. However, even if the 15% difference between the zero-span tensile strengths of Pulps 0 and 74 is attributed entirely to an actual decrease in the strength of the fibers, it is still substantially less than the difference found between the corresponding tensile strengths.*

Because the elastic modulus is measured from that part of the load elongation curve which is established in a relatively short time and at a relatively low strain, it is expected that interfiber bond rupture would play a lesser role than in the determination of the tensile strength. This is seen to be the case, where the change in modulus from Pulp 0 to Pulp 74 is only 27% compared to the 37% noted for the tensile strength.

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*Possible differences in extent of fiber-to-fiber bonding between handsheets prepared from the various treated and untreated pulps had been anticipated in the early phases of this work. It was planned to determine handsheet bonded areas by the light-scattering method (25-27). Since the effects of ethylamine treatment on the light scattering-surface area relationship were unknown, individual relationships for each pulp were to be established by correlation of light-scattering data with nitrogen gas adsorption data (25, 97). However, investigations by this worker and by members of the Institute Physics and Physical Chemistry Groups indicated that the low-temperature, high-vacuum gas adsorption tests may alter the amount of bonding existing in the specimens at normal room conditions. In addition, it appeared that the effect was a function of the degree of fiber-to-fiber bonding. Such behavior would, of course, invalidate any attempt to establish handsheet bonded and unbonded areas by low-temperature, high-vacuum gas adsorption techniques. Further studies of this problem are being conducted at the present time.

The relatively small differences between the tensile strengths and moduli of Pulps 74, 79, and 94 are somewhat unexpected in view of the continued decrease in pulp surface areas with increased ethylamine concentration (see Fig. 8). However, it is suggested that this may be due to an increasing fiber flexibility with decrease in crystallinity, which tends to allow better contact and bonding between fibers, thus offsetting partially the effects of the decreased surface area.

FIRST CREEP

The tensile creep test involves a study of the time-deformation relationship of a specimen supporting a constant tensile load. When the specimen is first subjected to the load, the resulting time-deformation relationship is referred to as first creep; if, after removal of the load and the passing of some arbitrary period of time, the load is reapplied, this resulting time-deformation relationship is called second creep, and so on. In an analogous manner, the period of recovery immediately following the first-creep test is referred to as the first-recovery test; after the second-creep test, the second-recovery test.

Because of the marked differences in rates usually observed in these tests at short and long times, the measured deformations are usually plotted versus the logarithm of the time. In this study, all tests were ended after 24 hours (86,400 sec.), unless terminated by rupture. The individual time-deformation data for all creep and recovery tests are given in Appendix II.

In any particular creep or recovery test, the type and extent of response are dependent upon the temperature, the relative humidity, and

the load applied. In all cases these factors must be specified. In this investigation, all tests were conducted at a temperature of 73°F. and a relative humidity of 50%.

In the creep and recovery tests conducted here, all loads were expressed as an apparent initial stress, in units of kg./sq.mm. The load, in kg., included the applied load and that contributed by the lower jaw assembly. The area, in sq.mm., refers to the calculated cross-sectional area of ovendry cellulose, perpendicular to the direction of the test. The stress must be referred to as apparent and initial since the actual number and area of elements supporting the applied load is unknown, and may be altered by the load or time.

In general, the effect of increased load during the creep test is to increase the magnitude and rate of creep. Brezinski (4) has suggested that the main effect of increased load is to shift the response toward earlier times. That is, the creep behavior observed under a particular load is the same as that which would have been observed at some later time under a lower load. As evidence, he showed that wood alpha-pulp handsheets followed the concept of the master creep curve (98). Schulz (38) has demonstrated that variations in the degree of wet-straining of handsheets have an effect on creep behavior similar to that of variations in applied stress; that is, the response to load is merely shifted in time.

In the master-creep-curve technique, the deformations observed at various loads were first reduced by a factor of the apparent initial stresses. The resultant curves were then shifted along the log-time

axis until they fit together in the regions of overlap, resulting in a single curve which hypothetically covered an extremely large time range. Because of the good precision obtained by Brezinski in fitting the curves in the regions of overlap he came to the conclusion mentioned above.

THE EFFECTS OF ETHYLAMINE TREATMENT ON FIRST-CREEP BEHAVIOR

Figures 14, 15, and 16 show the first-creep curves for Pulps 0, 74, and 94, respectively, at several different apparent initial stresses. These tests are for handsheets wet-pressed at 50 p.s.i. Similar families of curves were obtained with handsheets prepared from Pulps 60 and 79. The apparent initial stresses used were generally less than 75% of the load-elongation breaking stresses. All the tests were continued for 24 hours (86,400 sec.) except that at an apparent initial stress of 3.50 kg./sq.mm. shown in Fig. 16, which ruptured at about 300 sec.

It may be seen in Fig. 14, that, as the stress increased, the creep response of Pulp 0 handsheets tended to become linear with the logarithm of time. Brezinski (4) observed similar behavior with wood alpha-pulp handsheets and termed the linear portion logarithmic creep. Pulp 60 was found to follow this same pattern.

In the case of Pulps 74, 79, and 94, however, no such behavior was observed. At higher initial stresses, the deformation-log time relationships continued to exhibit curvature up to the point where the test was terminated, either by rupture or by reaching the end of the 24-hr. test period. This may be seen in Fig. 15 and 16. The significance of this will be considered later.

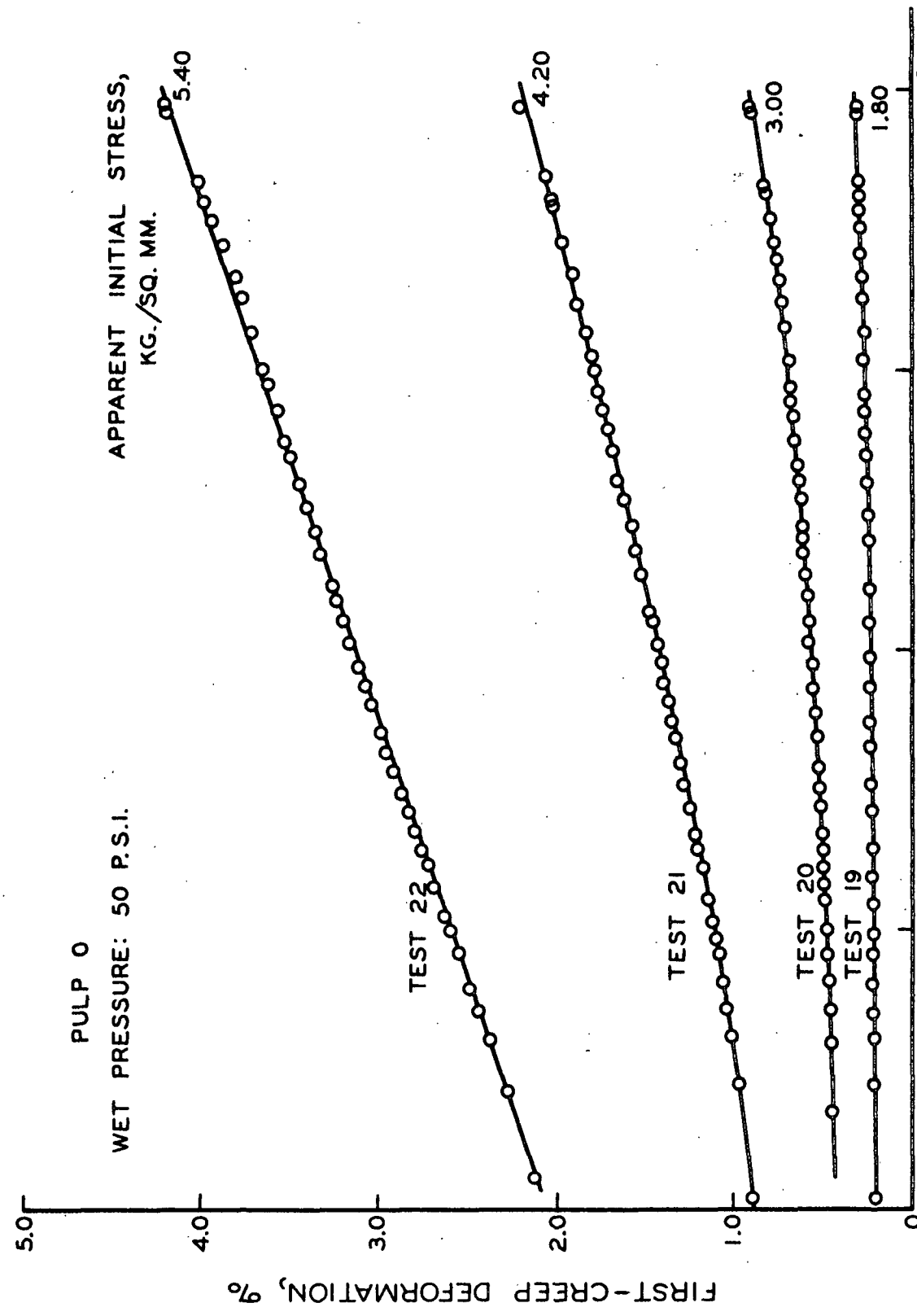

Figure 14. First-Creep Curves of Pulp O Handsheets at Different Apparent Initial Stresses

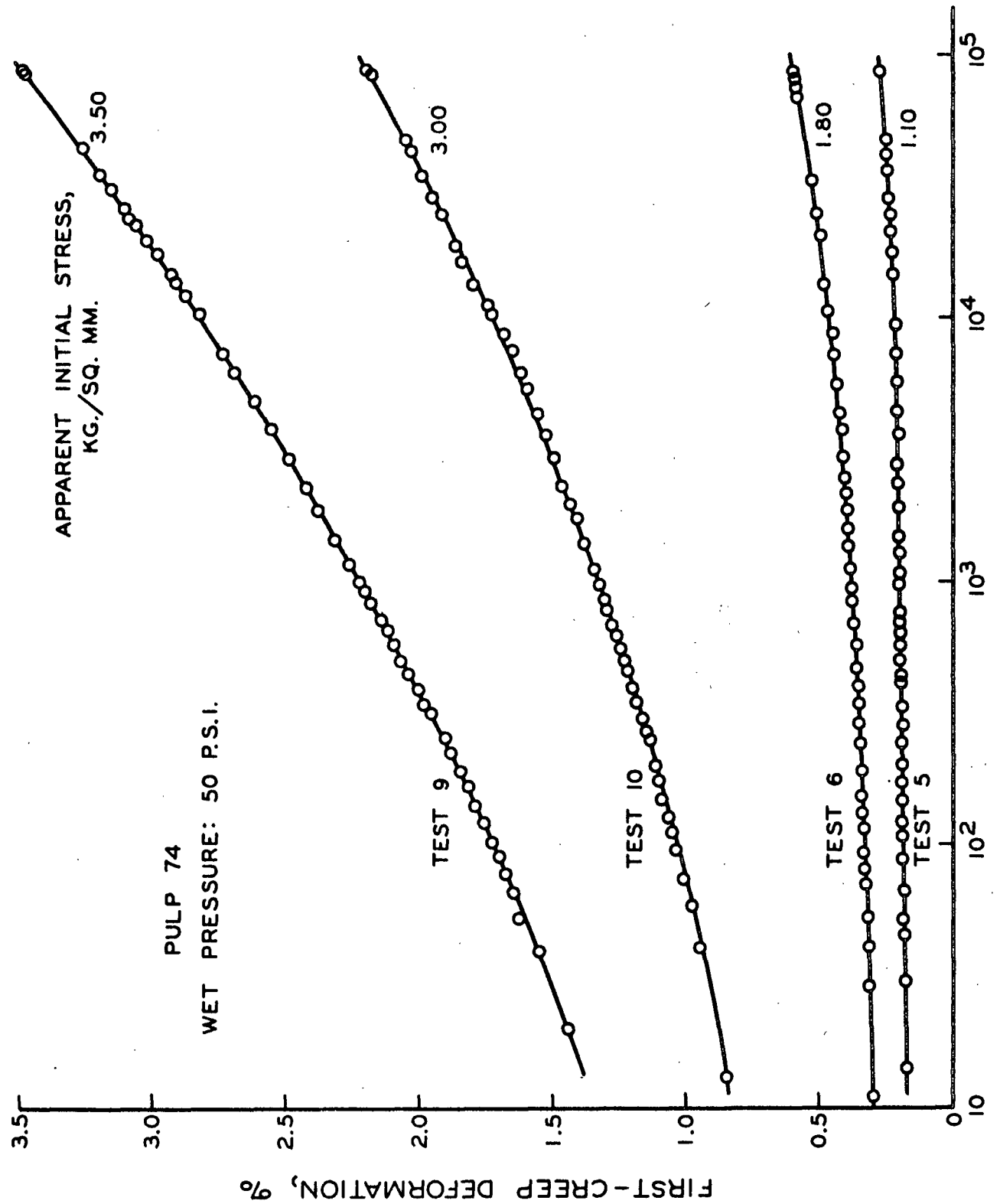

Figure 15. First-Creep Curves of Pulp 74 Handsheets at Different Apparent Initial Stresses

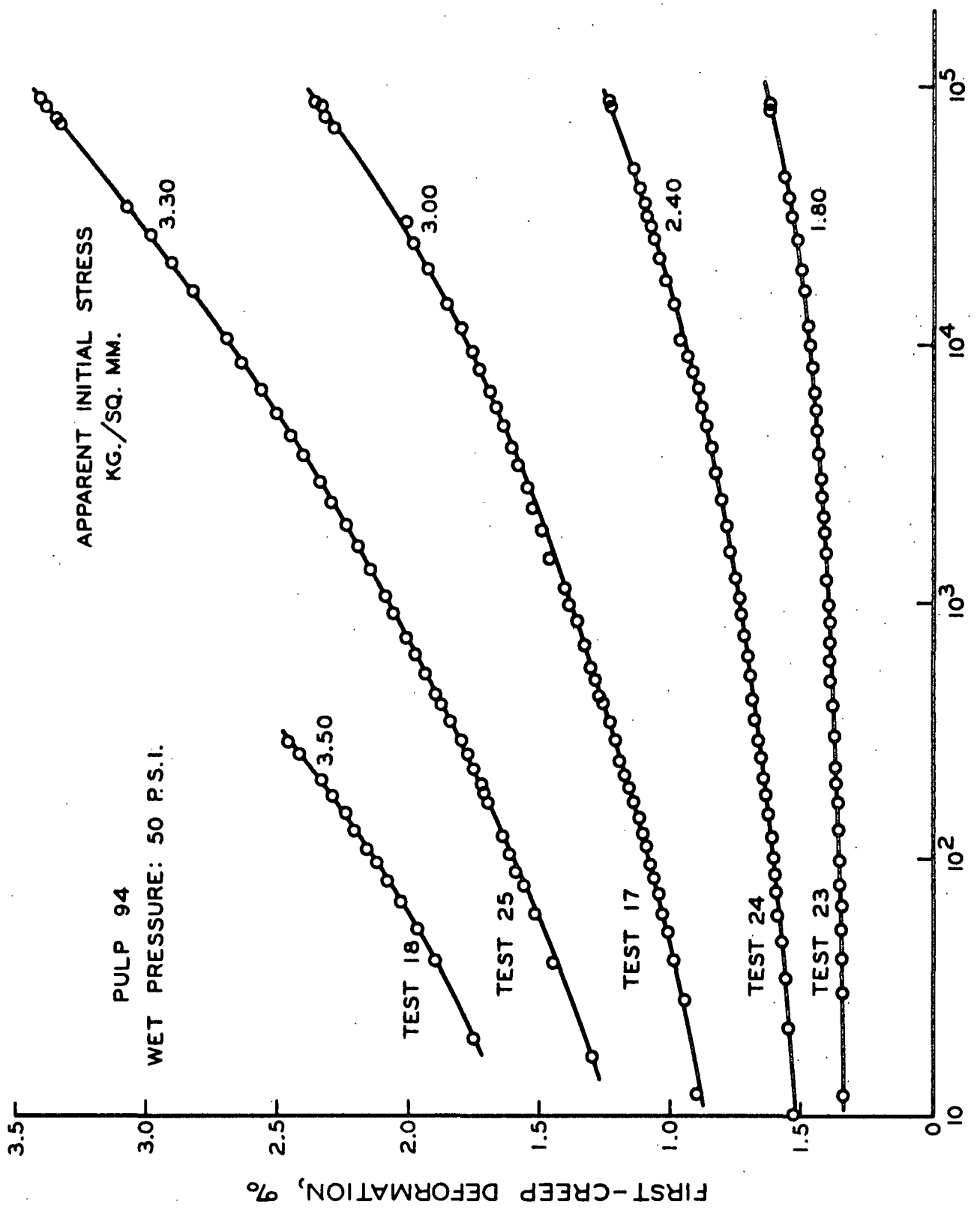

Figure 16. First-Creep Curves of Pulp 94 Handsheets at Different Apparent Initial Stresses

In Fig. 17 are shown the reduced creep curves of the tests from Pulp 0 shown in Fig. 14. The deformations for each curve have been divided by the appropriate values of the apparent initial stress and the resultant values have been termed the reduced deformations, in units of sq.mm./kg. From these curves, the master creep curve shown in Fig. 18 was constructed by shifting the curves along the log-time axis until they fit in the regions of overlap. (The abscissa in Fig. 18 is the log-time scale of the test at 1.80 kg./sq.mm.) As found by Brezinski (4) and Schulz (38), the shift in log time necessary to fit the curves together and form the master creep curve was a linear function of the initial stress. In the case of Pulp 0 this shift was 2.01 decades of log time per unit initial stress.

In the case of Pulps 74, 79, and 94, however, attempts to construct master creep curves were unsuccessful. The creep deformations at all stresses tended to be too high at long times to permit proper fitting of the curves. This can be seen most clearly in the case of Pulp 94, Fig. 16. If these increases in response at long times were ignored and a "pseudo-master" creep curve was formed, it was found that the required shifts in log time were not a linear function of apparent initial stress, but increased sharply at the high stresses.

Brezinski (4) found that the ability to form a master creep curve was not affected by the extent of interfiber bonding. Furthermore, Schulz (38) has shown that for any degree of handsheet wet-straining, which is believed to alter the number and distribution of elements supporting the load (an interfiber effect), master creep curves may be constructed.

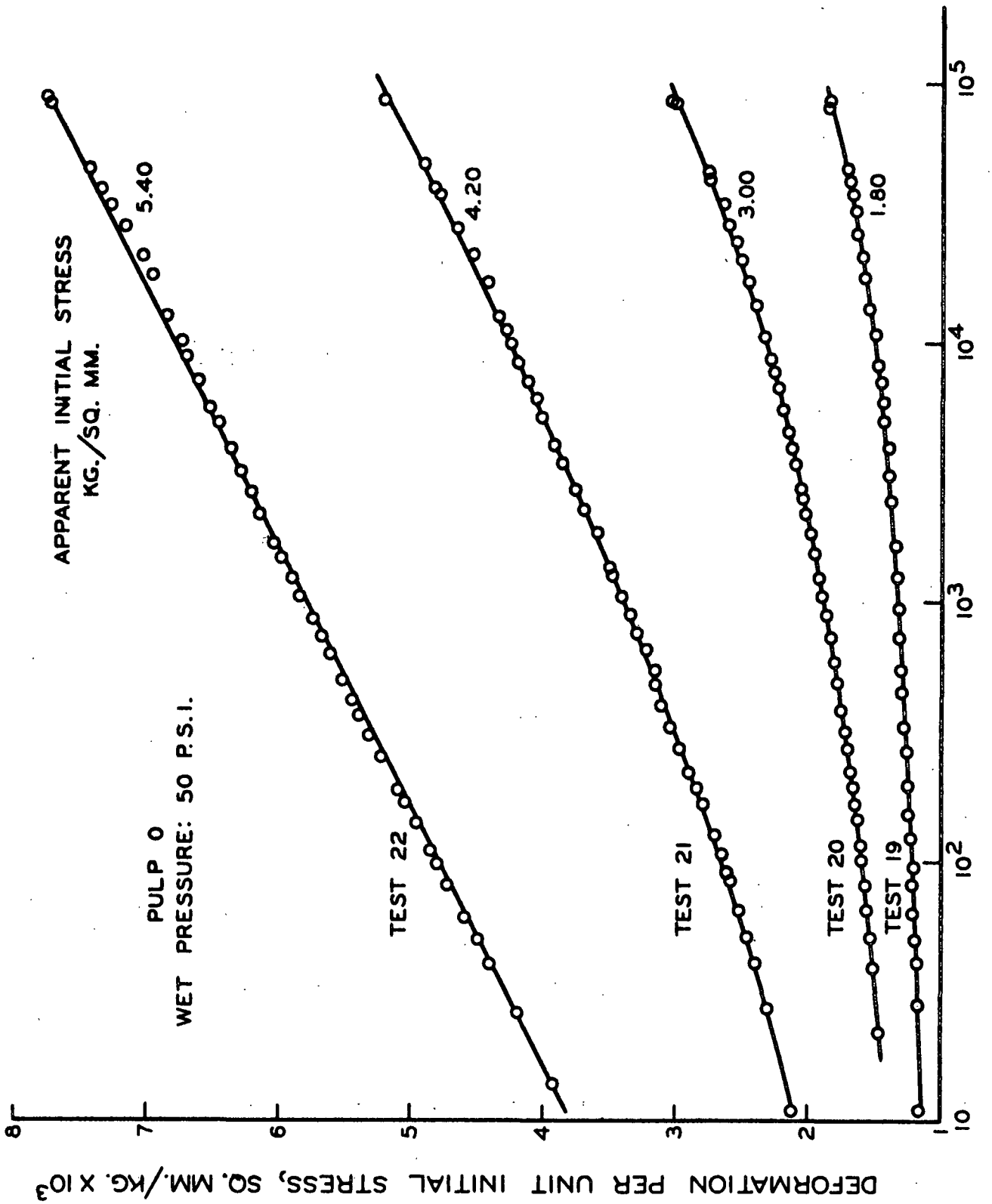

Figure 17. Reduced First-Creep Curves of Pulp O Handsheets

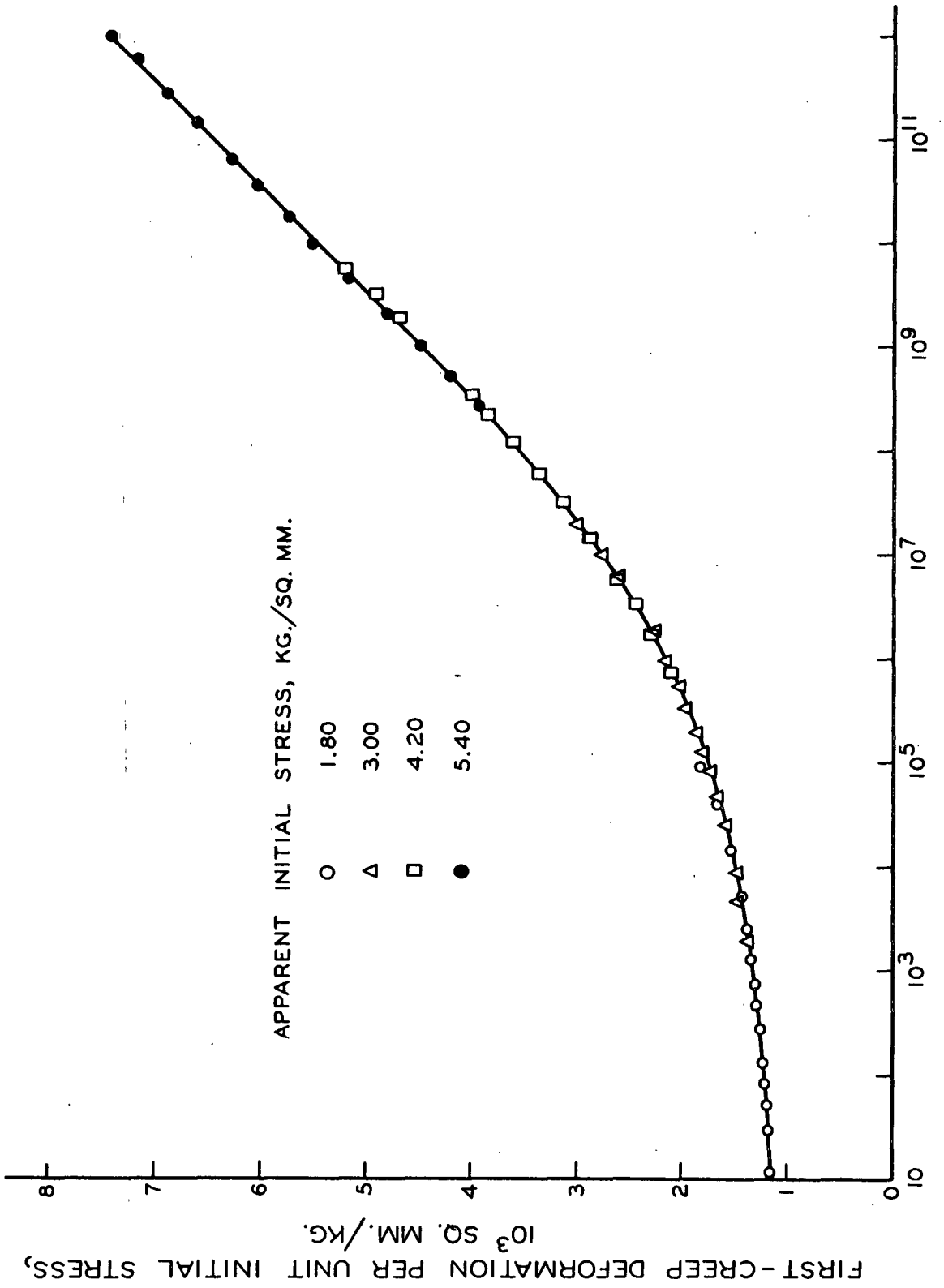

Figure 18. Master Creep Curve of Pulp O Handsheets at 50 p.s.i. Wet Pressure

Brezinski (4) and Schulz (38) have both suggested that creep observed at long times (at relatively low loads) or at high stresses (at relatively short times) is largely nonrecoverable. Considerable evidence has been advanced to support this contention. Whether these mechanisms are related to intrafiber, molecular properties, or to interfiber, macroscopic phenomena, however, has been disputed. Whereas Brezinski (4) supports the former view, Rance (3) and Sanborn (19) have both hypothesized that the recovery is retarded by interfiber frictional effects which simulate molecular viscoelastic behavior.

However, the inability of the decrystallized specimens studied here to fit the master creep curve concept offers distinct evidence that at least a portion of the nonrecoverable creep is related to intrafiber mechanisms, and that this nonrecoverable creep is reduced as the concentration of the decrystallizing solutions is increased. Additional evidence to support this view will be presented later.

In Fig. 19 and 20 are shown the first-creep curves of all five pulps at two levels of apparent initial stress--1.80 and 3.00 kg./sq.mm., respectively.*

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*In the course of this study, it was found that the observed creep response of a handsheet was influenced by the moisture content of the blotters used in its preparation. Consequently, all the test sheets used to give the data shown in Fig. 19 and 20 were prepared with blotters conditioned at 50% R.H. In all other cases, comparisons were made only between sheets prepared from Pulps 0, 74, and 94. These latter sheets were prepared within a short period of time at conditions which were unknown, but sufficiently constant to allow valid comparisons. A more detailed examination of this phenomenon is given in Appendix A.

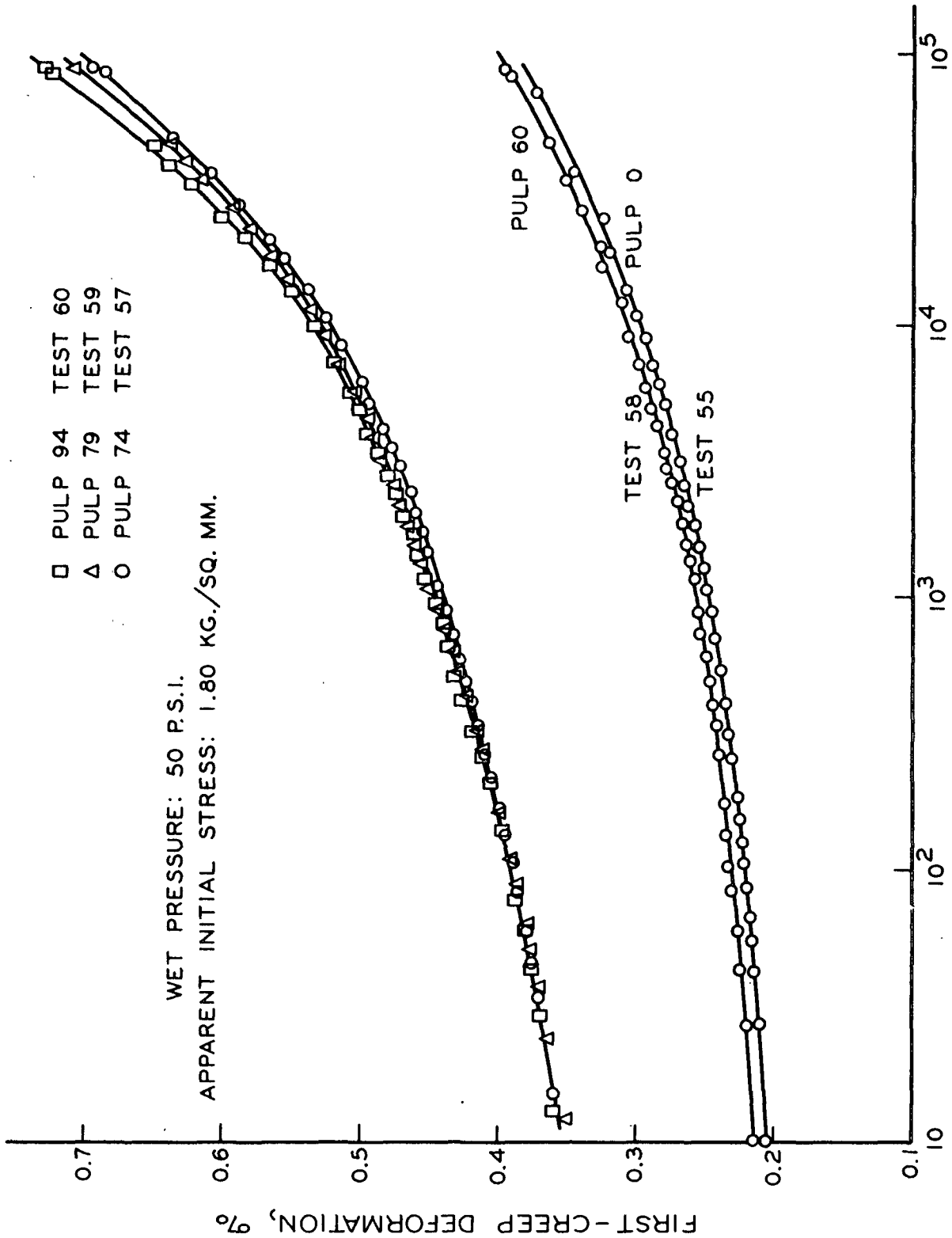

Figure 19. First-Creep Curves of 50-p.s.i. Handsheets at 1.80 kg./sq.mm. Initial Stress

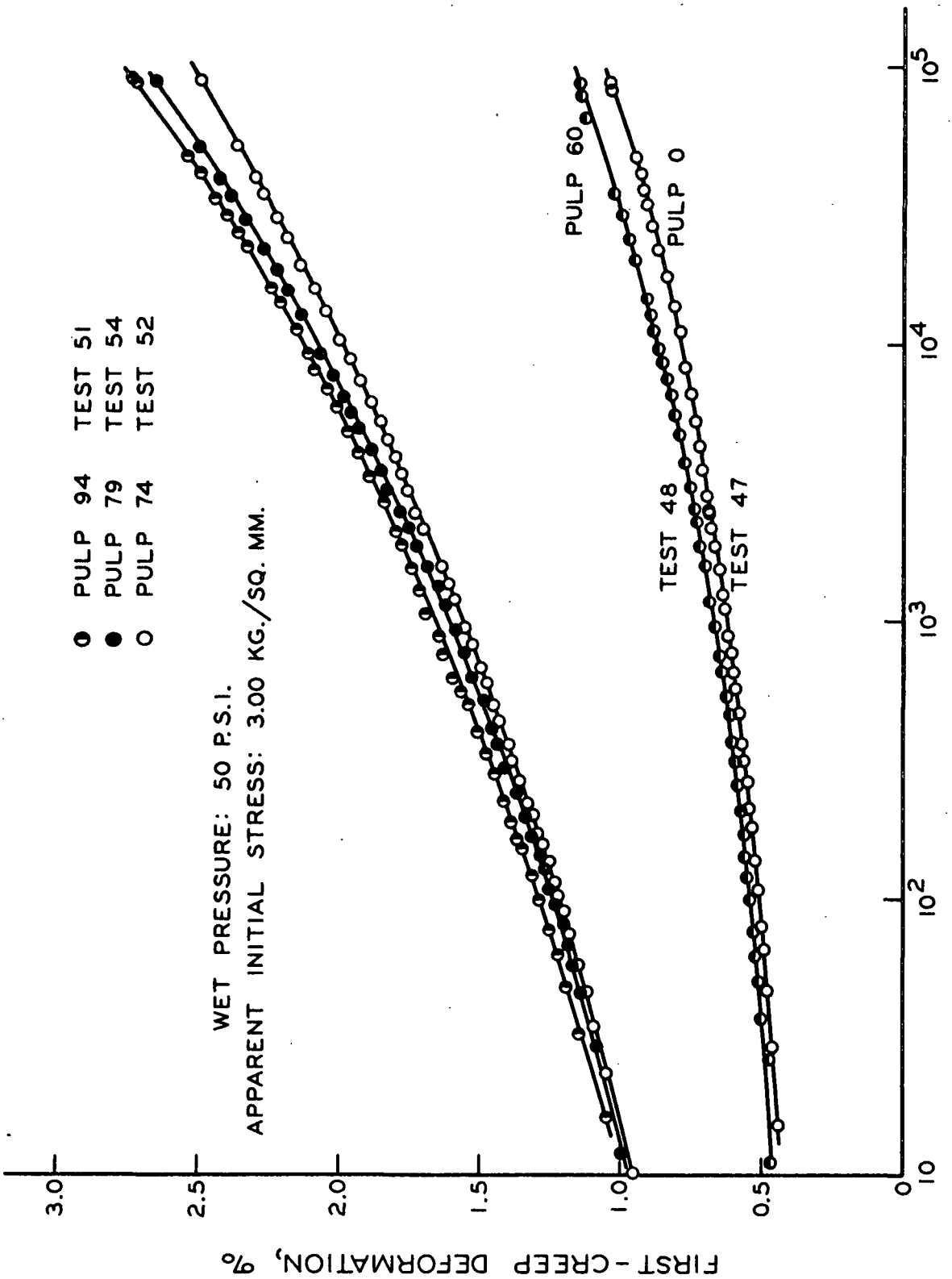

Figure 20. First-Creep Curves of 50-p.s.i. Handsheets at 3.00 kg./sq.mm. Initial Stress

The similarity between the behavior shown in Fig. 19 and 20 and the results of the load-elongation tests described earlier is obvious; there is a marked steplike change in the response to stress. Again, it is suggested that this sudden change upon going from the crystalline pulps, 0 and 60, to the decrystallized pulps, 74, 79, and 94, is caused primarily by the reduction in pulp surface areas, resulting in a decrease in handsheet fiber-to-fiber bonding at similar conditions of handsheet preparation. In view of the similarity in behavior of Pulps 74, 79, and 94, only a small fraction of this difference between the crystalline and decrystallized pulps can be attributed to an alteration of intra-fiber structure. However, some of it may be due to a real decrease in fiber strength with decrystallizing treatment, as well as to the loss of interfiber bonding.

Schulz (38) has shown that increased wet-straining of handsheets causes substantial reduction in creep response and has suggested that this is caused by an improvement in the internal stress distribution. As the number of load-carrying elements within the sheet is increased, each element supports a smaller fraction of the total load, the effective stress on each is decreased, and the resultant deformation of the entire specimen is reduced. Conversely, any factor such as decreased pulp surface area, tending to reduce the bonding within the sheet causes an increase in the creep strain of the entire specimen at comparable levels of applied stress. It will be seen later that variations in wet pressure have a similar effect.

While it is tempting at this point to attribute the small differences in creep behavior of Pulps 74, 79, and 94 to differences in fiber crystallinity, it is equally probable that they were caused by differences in fiber-to-fiber bonding. Indeed, the differences between Pulps 0 and 60 seen in Fig. 19 and 20 were caused quite probably by this source alone.

THE EFFECTS OF WET PRESSURE ON FIRST-CREEP BEHAVIOR

Figure 21 shows the first-creep curves of handsheets prepared from Pulps 0, 74, and 94, wet-pressed at 250 p.s.i., and tested at an apparent initial stress of 3.00 kg./sq.mm. Figure 22 shows the corresponding curves from handsheets wet-pressed at 1000 p.s.i. By comparing these curves to those in Fig. 20 (note that the ordinates in Fig. 21 and 22 have been expanded), it can be seen that the creep of all three pulps has been substantially reduced by the higher wet pressures. The reasons for this behavior have been stated above. However, the important things to note here are the relationships between the three curves of each figure as the wet pressure is increased.

The difference between the total creep in 24 hr. of Pulps 0 and 74 has been reduced from about 140% at 50 p.s.i. to about 70% at 250 p.s.i. At 1000 p.s.i., this difference has been further reduced to about 57%. In following the same steps, the difference between Pulp 74 and Pulp 94 was first reduced from about 9 to about 5%, and then increased to approximately 13% at 1000 p.s.i. wet pressure.

If the manner in which the sheet responds to a tensile load were controlled entirely by interfiber mechanisms, such dependence of the

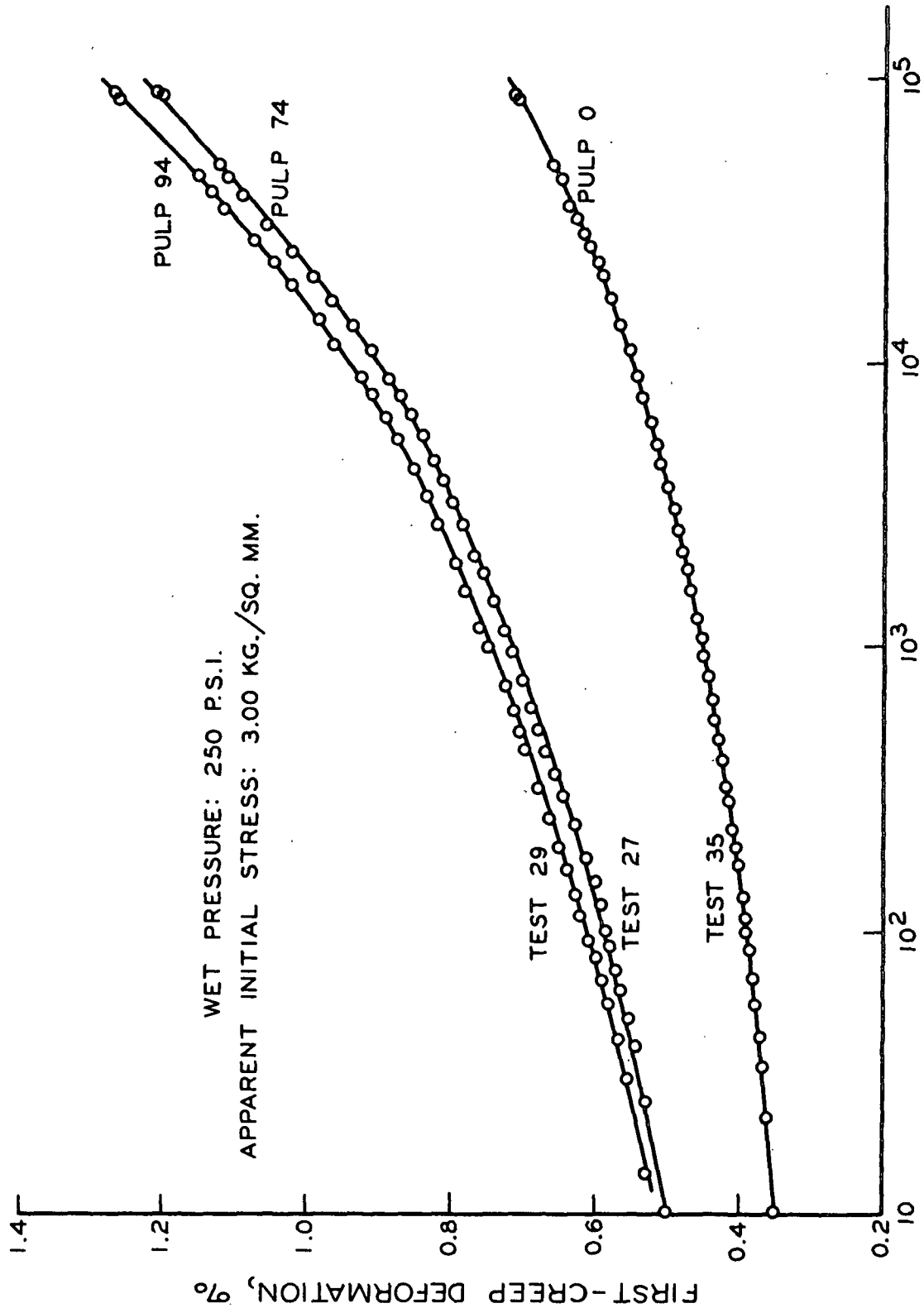

Figure 21. First-Creep Curves of 250-p.s.i. Handsheets at 3.00 kg./sq.mm. Initial Stress

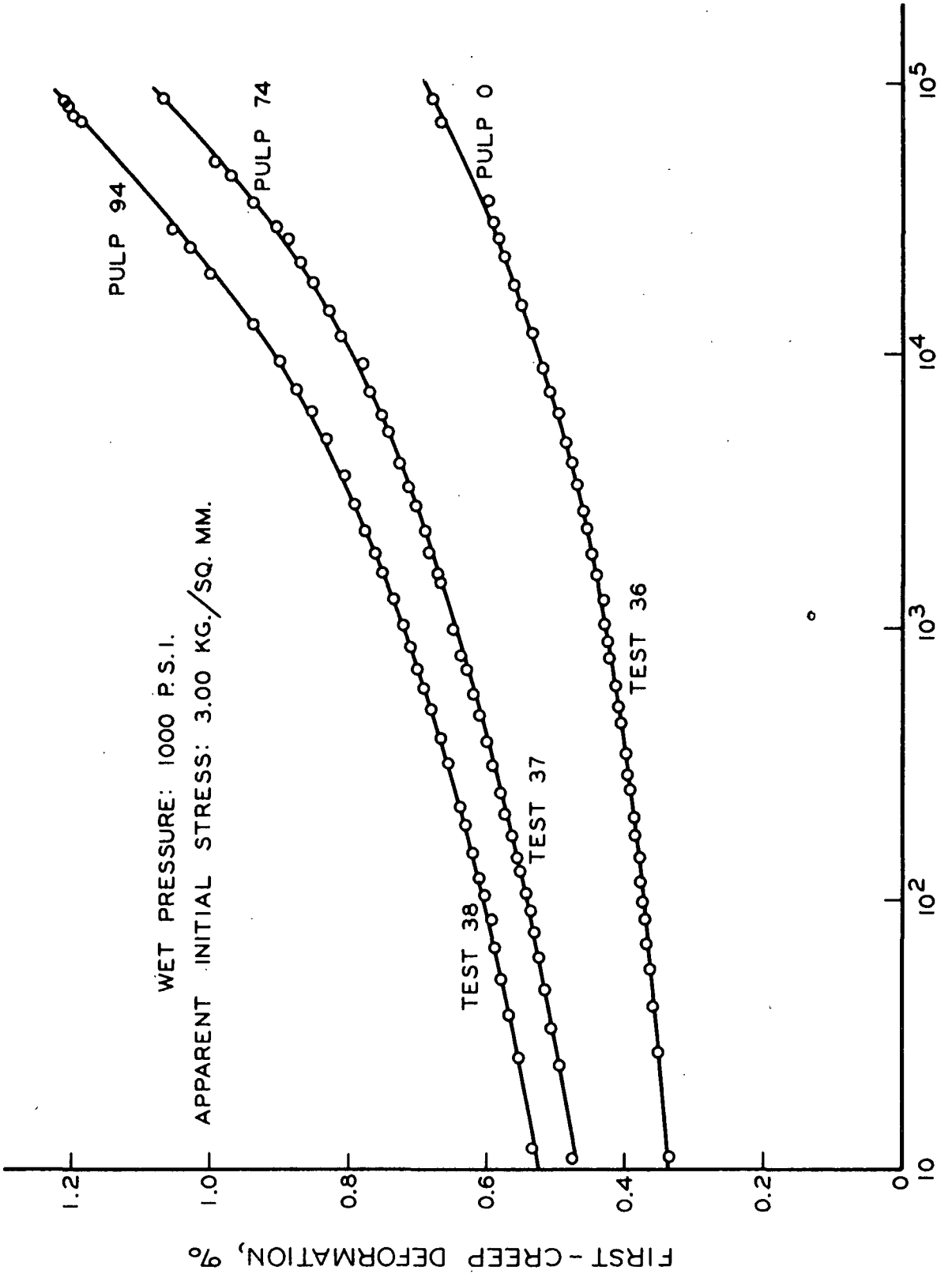

Figure 22. First-Creep Curves of 1000-p.s.i. Handsheets at 3.00 kg./sq.mm. Initial Stress

relative creep of the three pulps on wet pressure would not be expected. That is, if interfiber mechanisms alone were controlling, the creep of all three pulps would continue to converge as the wet pressure was increased, for at the higher pressures, differences between the degrees of fiber-to-fiber bonding would become less and less important. However, this is not found to be the case with Pulps 74 and 94. At 1000 p.s.i. wet pressure the separation between them was greater than that at 250 and 50 p.s.i.

These data suggest that both interfiber and intrafiber mechanisms play a part in controlling the response of the paper to stress, and that the relative importance of each depends upon the over-all strength of the sheet. As the degree of fiber-to-fiber bonding is increased, more structural units join in sharing the load, and more of the strain response is from that of the fibers. Thus, the properties of the fibers themselves, rather than merely their ability to form bonds, become more important. The extreme case of such fiber-to-fiber bonding would be a glassine sheet in which the transition from fiber to fiber is almost indistinguishable, resulting in a situation in which almost the entire strain results from fiber deformations.

Van den Akker, et al. (6) studied the number of fibers broken in the zone of rupture of tensile tests. They found that the number broken increased substantially as the degree of bonding increased and thus reached a conclusion similar to that given above. Sanborn (19), after a study of the energy absorption of paper in tensile creep tests, suggested that the main portion of the energy loss was absorbed in the rupture of fiber-to-

fiber bonds. However, he also hypothesized that appreciable amounts of energy might be absorbed by the fiber elements within the sheet during deformation.

Apparently, in the case of Pulps 0 and 74, the differences in degree of interfiber bonding were great enough, and the differences in intrafiber properties small enough, that the curves continued to converge as the wet pressure was increased. That is, the disparity between the degrees of interfiber bonding tended to overshadow any effects which might have resulted from differences in intrafiber, molecular structure. However, with Pulps 74 and 94, the differences in interfiber bonding appear to have been substantially eliminated at 250 p.s.i. wet pressure, so that the effects of variation in intrafiber properties could be observed. Additional evidence will be presented in the considerations of creep recovery and the relationship between creep and recovery.

CREEP RECOVERY BEHAVIOR

When the stress in a creep test is removed from the specimen after some arbitrary period of time, the specimen will recover a portion of the deformation caused by the creep stress. The extent of recovery will depend upon the conditions of time, temperature, and relative humidity. When making comparisons between creep and recovery, these conditions are generally held constant.

Figure 23 shows the first-recovery curves of Pulps 0, 74, 79, and 94 from 50-p.s.i. handsheets tested at an apparent initial stress of 3.00 kg./sq.mm. These are the recovery curves of the tests shown in Fig.

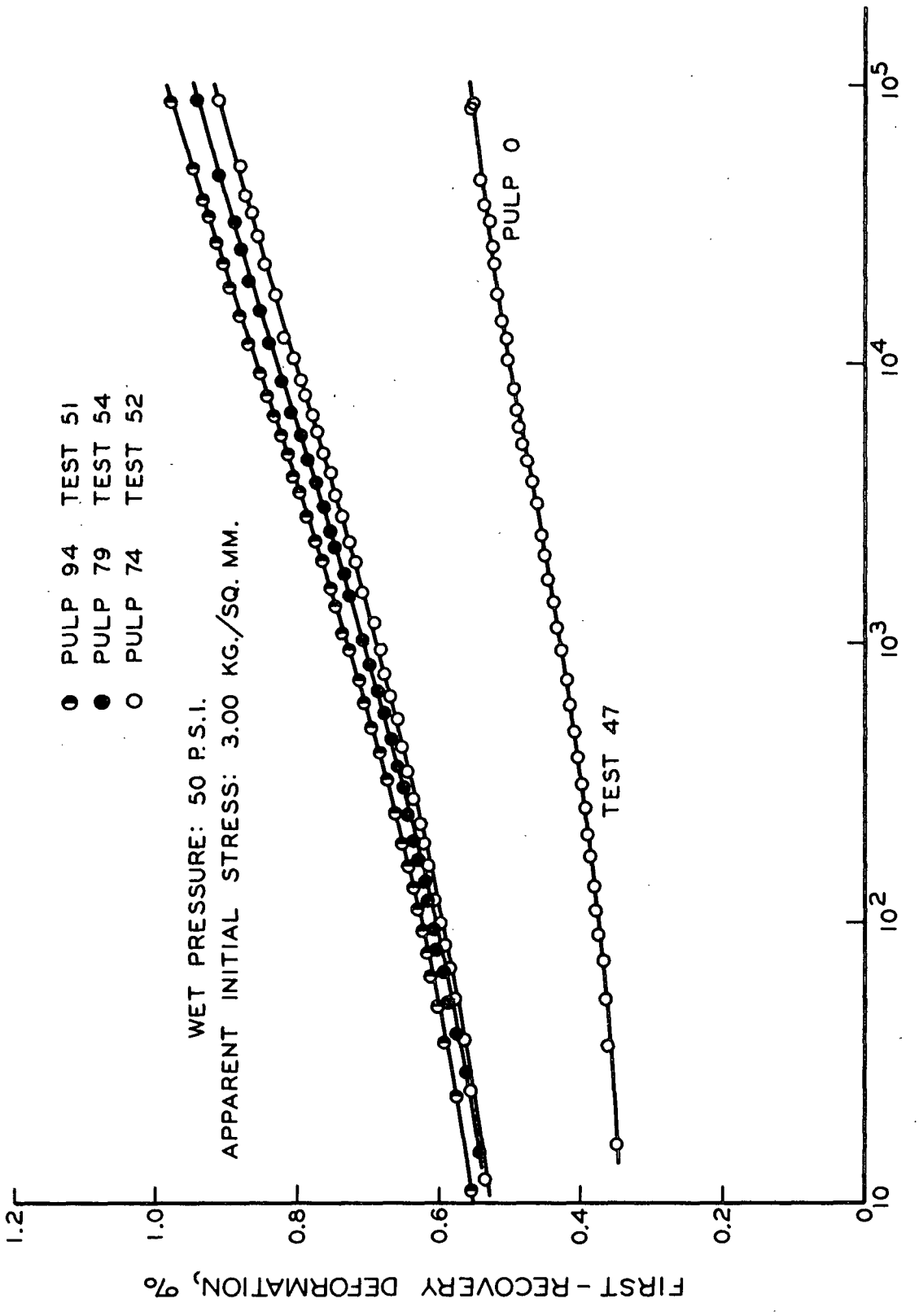

Figure 23. First-Recovery Curves of 50-p.s.i. Handsheets at 3.00 kg./sq.mm. Initial Stress

20. In Fig. 24 and 25 are the corresponding curves for Pulps 0, 74, and 94 from handsheets wet-pressed at 250 and 1000 p.s.i., respectively. In Table VI are listed the differences in 24-hr. recovery deformations between Pulps 0 and 74 and between Pulps 74 and 94 at the three wet pressures.

TABLE VI

DIFFERENCES IN 24-HOUR RECOVERY DEFORMATIONS BETWEEN PULPS 0 AND 74 AND BETWEEN PULPS 74 AND 94[a]

Wet Pressure, p.s.i.	Pulps 0 and 74	Pulps 74 and 94
50	67%	7.7%
250	33	7.2
1000	33	16.6

[a]Each difference is calculated as a percentage increase over the lower deformation.

It can be seen from the figures and from Table VI that as the wet pressure was increased from 50 to 250 p.s.i. the differences between Pulps 0 and 74 decreased sharply in a manner similar to that of the first-creep curves. However, as the wet pressure increased to 1000 p.s.i., the difference, in this case, did not decrease further but remained essentially constant. With Pulps 74 and 94, the behavior was similar to that of the creep curves, decreasing first and then increasing.

It is interesting to note the spacing of the recovery curves shown in Fig. 25 as contrasted to that in Fig. 23 and 24. Although it is

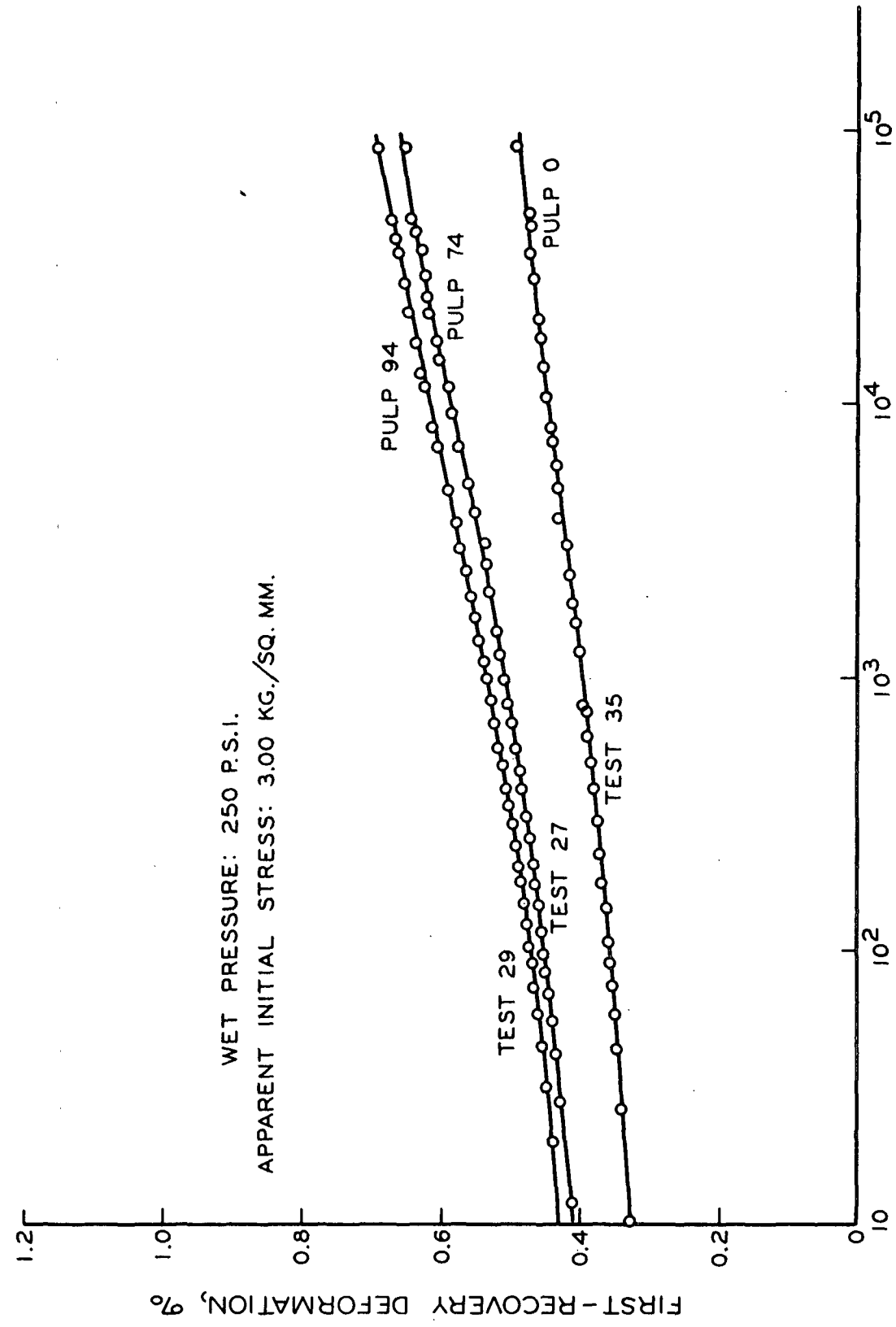

Figure 24. First-Recovery Curves of 250-p.s.i. Handsheets at 3.00 kg./sq.mm. Initial Stress

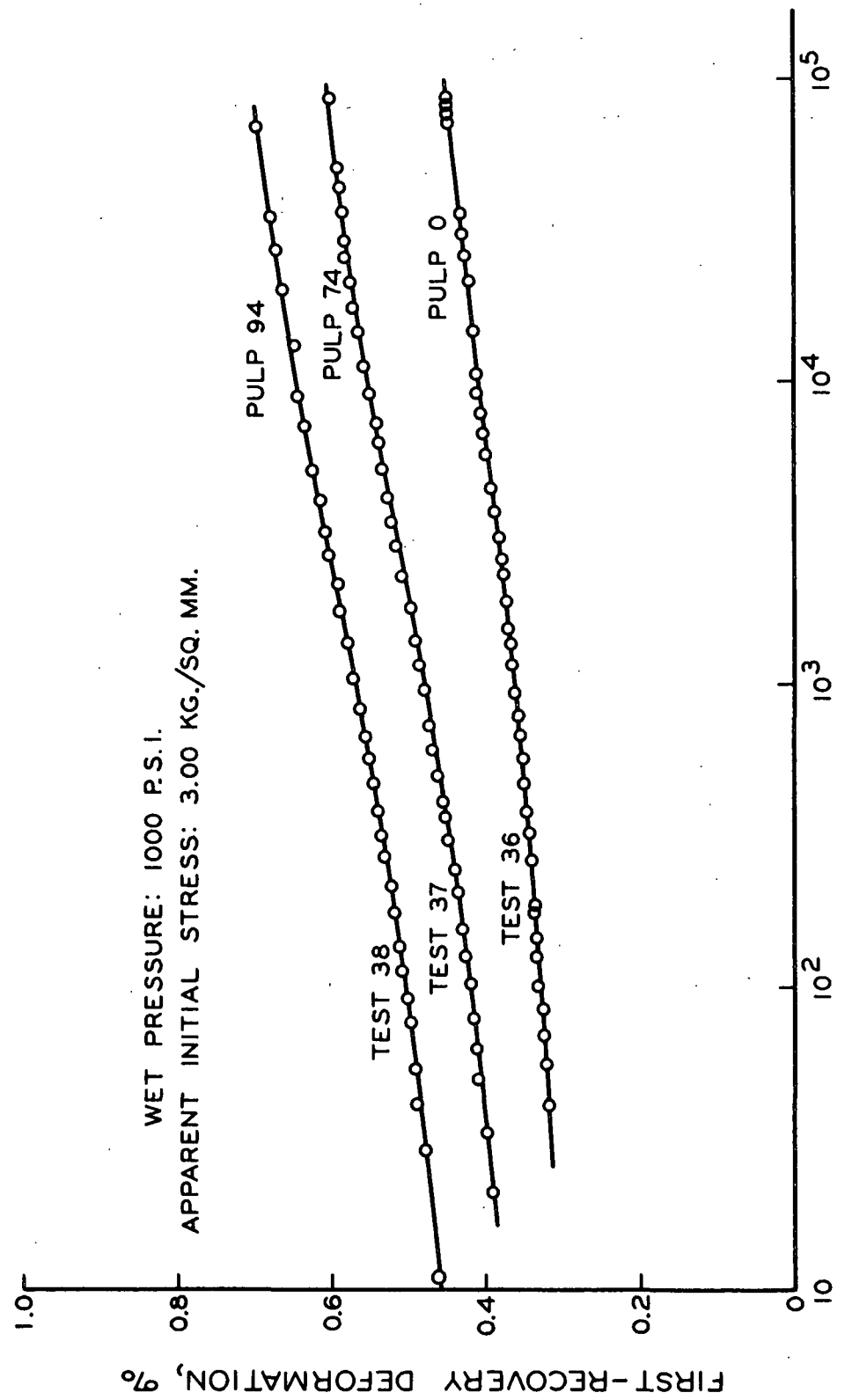

Figure 25. First-Recovery Curves of 1000-p.s.i. Handsheets at 3.00 kg./sq.mm. Initial Stress

expected that the recovery curve for Pulp 74 would be even closer to that of Pulp 0 if intrafiber properties were the only controlling factor in recovery, the fact that it has tended to approach this point, while diverging from that of Pulp 94, is felt to be significant. It suggests that at this high wet pressure the differences in handsheet fiber-to-fiber bonding between the three pulps have been largely eliminated and that the recovery, even more than the creep, is primarily controlled by intrafiber mechanisms.

Thus, as the severity of the decrystallizing treatment is increased the recovery is increased. This behavior is magnified at higher levels of interfiber bonding where there is a tendency for the strains within the fibers to increase. At lower levels of bonding, the effects are partially overshadowed by differences in the extent of bonding.

THE RELATIONSHIP BETWEEN CREEP AND RECOVERY

In view of the fact that it was not possible in the work discussed above, to separate completely the effects of inter- and intrafiber properties on creep behavior, attention was directed to the relationship between creep and recovery. This relationship has been studied and discussed by both Schulz (38) and Brezinski (4). Brezinski found that a unique relationship existed between total first-creep deformation in a given period of time and total first-recovery deformation in an equal period of time. This function was unaffected by either the applied stress or the extent of fiber-to-fiber bonding.

In a similar manner, Schulz (38) showed that the creep-recovery function was also unaffected by the degree of wet-straining and time,

provided the periods of creep and recovery were equal. Any observed recovery deformation was determined only by the amount of creep preceding it, and was unrelated to the path by which the particular creep deformation was reached. Thus, a given total first-creep deformation, whether achieved by a short-term test at high stress or a long-term test at low stress, yielded the same amount of recovery in an equivalent period of time.

Brezinski (4) concluded from this and from additional evidence that the mechanisms of recoverable and nonrecoverable creep were not unrelated, and that such mechanisms were most probably of an intrafiber, molecular nature.

Schulz (38) had hypothesized that one of the possible effects of wet-straining might be a reduction in handsheet capacity for nonrecoverable creep, i.e., it might act as a form of mechanical conditioning. However, as noted above, this was not found to be the case.

In view of these findings, it was felt that this creep-recovery relationship might provide a means of separating inter- and intrafiber variables, since it had been shown that wide variations in interfiber properties had little or no effect upon it. Consequently, a series of tests were conducted in which specimens from Pulps 0, 74, and 94, at all three wet pressures, were subjected to several different stresses, and their creep and recovery deformations in 24 hours were measured. Table VII lists the wet pressure, apparent initial stress, and the total first-creep and total first-recovery strains for each test. These data are plotted in Fig. 26.

TABLE VII

TOTAL FIRST-CREEP AND TOTAL FIRST-RECOVERY DEFORMATIONS AT VARIOUS WET PRESSURES AND INITIAL STRESSES[a]

Pulp	Test No.	Wet Pressure, p.s.i.	Apparent Initial Stress, kg./sq.mm.	Total Creep Deformation, %	Total Recovery Deformation, %
0	19	50	1.80	0.331	0.253
	20	50	3.00	0.906	0.525
	21	50	4.20	2.190	0.833
	22	50	5.40	4.183	1.089
	68	50	3.60	1.399	0.695
	35	250	3.00	0.713	0.491
	39	250	1.80	0.260	0.212
	69	250	5.00	2.723	0.919
	70	250	6.00	4.183	1.107
	36	1000	3.00	0.680	0.453
	42	1000	1.80	0.287	0.235
74	5	50	1.10	0.270	0.211
	6	50	1.80	0.606	0.382
	8	50	3.02	2.199	0.885
	9	50	3.50	3.483	1.067
	10	50	3.00	2.189	0.896
	71	50	3.30	2.689	1.000
	7	250	3.47	1.942	0.810
	27	250	3.00	1.210	0.652
	40	250	1.80	0.384	0.313
	72	250	3.80	2.303	0.916
	73	250	4.10	3.001	1.057
	37	1000	3.00	1.067	0.602
	43	1000	1.80	0.380	0.289
94	16	50	1.80	0.647	0.470
	17	50	3.00	2.356	0.966
	23	50	1.80	0.627	0.412
	24	50	2.40	1.234	0.680
	25	50	3.30	3.401	1.118
	74	50	3.15	2.627	1.031
	29	250	3.00	1.266	0.690
	41	250	1.80	0.414	0.292
	75	250	3.80	2.649	1.035
	76	250	4.00	3.127	1.114
	38	1000	3.00	1.219	0.700

[a]Duration of each creep and each recovery test: 24 hr.

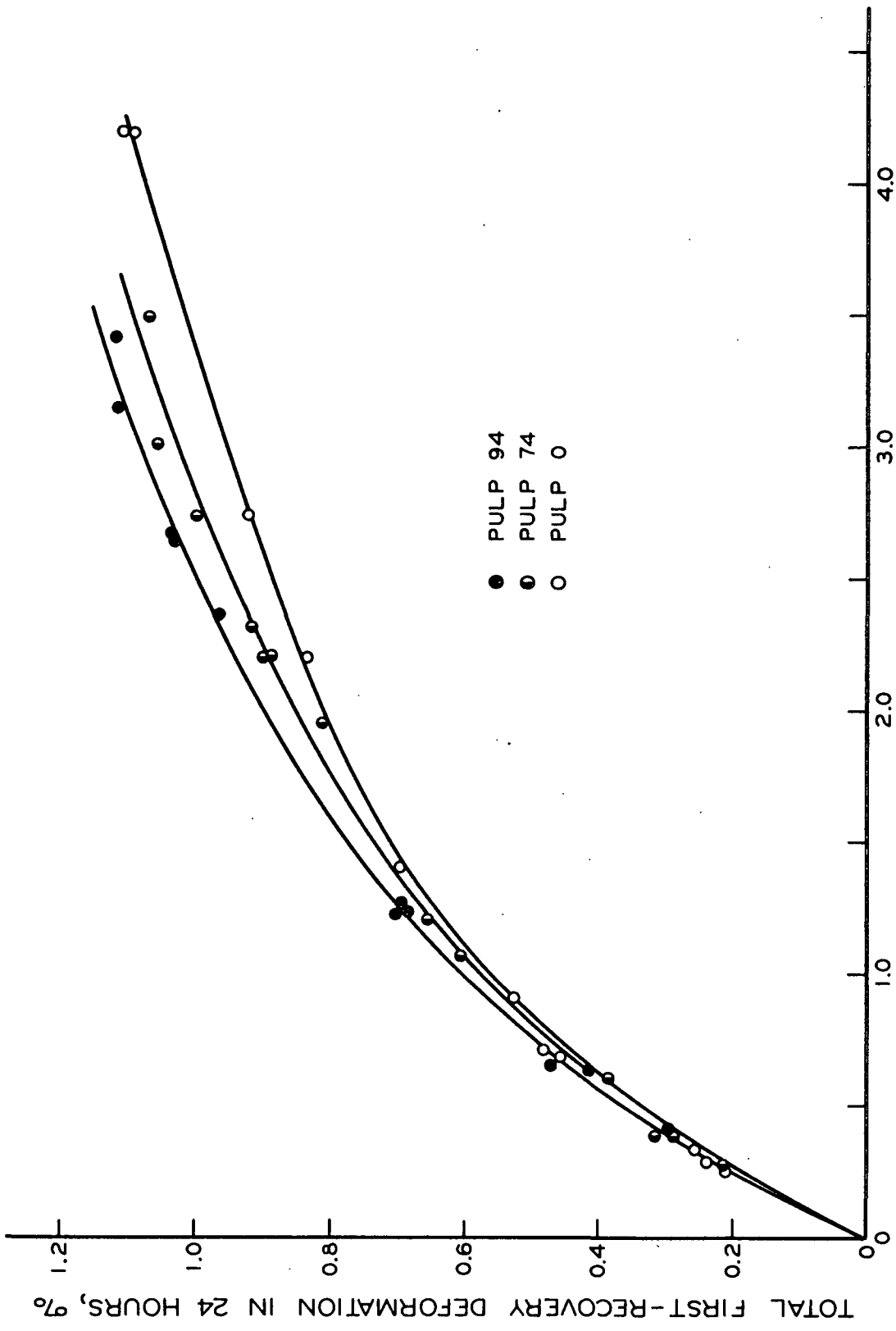

Figure 26. Total First-Recovery Deformation in 24 Hours versus Total First-Creep Deformation in 24 Hours

These curves are similar in shape to those reported by Brezinski (4) and Schulz (38). It is important to note that as the total first-creep strain is increased, the recoverable portion drops off quite rapidly. A creep deformation of 1% is about 60% recoverable while one of 3% is only about 30% recoverable.

However, the most important characteristic of Fig. 26 is the fact that three separate relationships between creep and recovery were found for the three pulps tested. As the concentration of ethylamine was increased, the extent of recovery for a given amount of first creep was also increased. As might be expected, the separation of the three curves became greater as the nonrecoverable deformation was increased. For a given pulp, the relationship was unaffected by either wet pressure or apparent initial stress.

The significance of these curves cannot be overemphasized, for they lend strong support to the hypothesis posed earlier that the alteration of intrafiber structure reduces the nonrecoverable portion of the first-creep strain. It may be recalled that this phenomenon was evidenced by the failure of the decrystallized pulps to follow the concept of the master creep curve. Because of excessive creep at long times or at high stresses, the creep curve at a particular stress could not be fitted to one at another stress by shifting the curve along the time axis. It was suggested that this was due to a reduction of the nonrecoverable portion of first creep, this portion usually being attributed to strains occurring at long times or at high stresses (38). The data of Fig. 26 support this view.

In general, recoverable deformations in polymeric materials are attributed to retarded elastic mechanisms. In amorphous polymers, the nonrecoverable strains can usually be related to displacements of entire molecules or to the formation of stable crystalline structures (7). However, in partially crystalline materials, such as cellulose, the former mechanism (displacement of entire molecules) is highly unlikely, for the probability of a molecule not passing through at least one region of high lateral order, and thus being restrained, is quite small (7).

Brezinski (4) has suggested that one of the mechanisms of nonrecoverable creep in paper may be the formation of additional crystalline material within the cellulose, under the influence of the applied stress. This structure may be permanent or may be stable only under the test conditions--that is, it may be a metastable structure which would be destroyed at some higher temperature or relative humidity, but which appears permanent at the conditions used in the test. Of course, some molecular arrangements of this type may also be completely reversible; when the stress is removed, this crystalline structure melts and the specimen exhibits elastic recovery. Rubber has been shown to behave in this manner (37).

It is suggested, therefore, that the mechanism by which the nonrecoverable creep is reduced may be the inhibition of the formation of metastable crystalline structures within the cellulose fibers while enhancing the formation of reversible structures. Since wide variations in interfiber properties have been shown to have an insignificant effect

upon the creep-recovery function, the behavior shown in Fig. 26 can be explained only on the basis of intrafiber, molecular mechanisms. Even if the decrystallizing treatment in ethylamine were to have some unusual effect upon the macrostructure of the fibers and the resultant handsheet, which would cause a shift in the creep-recovery relationship, one would not expect to observe the continuous change shown in Fig. 26. Rather, it is probable that one would obtain only two curves--one for Pulp 0 and one for Pulps 74 and 94, in a manner analogous to the abrupt changes in the other pulp and handsheet properties observed earlier.

On this basis, therefore, it must be concluded that intrafiber, molecular mechanisms do play an important role in the creep and recovery behavior of paper. However, in view of the dependency of this behavior on wet pressure, as described earlier, and in view of the work done by Sanborn (19) and by Van den Akker, et al. (6), it must also be concluded that the relative importance of this role, as compared to that of interfiber mechanisms, is highly dependent upon the over-all strength and structure of the specimens under examination.

SUMMARY AND CONCLUSIONS

A high-purity, muslin rag pulp was treated in various concentrations of aqueous ethylamine solutions to provide four different levels of fiber degree of crystallinity. Special equipment was designed and constructed which allowed treatment and washing of the pulp in a nitrogen atmosphere. Intrinsic viscosities, measured in cupriethylenediamine solution, showed no apparent decrease in the average degree of polymerization of the pulps.

Pulps were decrystallized in solutions of 74, 79, and 94% ethylamine. Each was labeled according to the concentration of solution in which it was treated. Thus, for example, the pulp treated in 74% aqueous ethylamine was labeled Pulp 74. The untreated pulp was labeled Pulp 0 for convenience. Another portion of pulp was treated in 60% ethylamine to act as a control. This concentration caused an insignificant decrease in crystallinity; similar results were found by Segal, et al. (62).

The extent of pulp decrystallization was determined by an x-ray diffraction technique (75) and a "crystallinity index" was calculated from the resulting intensity curves. Pulps 0, 60, 74, 79, and 94 were found to have crystallinity indices of 70.1, 69.4, 62.8, 56.0, and 52.1, respectively.

Pulp properties were characterized by measuring the Schopper-Riegler freeness, the filtration resistance, the specific surface, swollen specific volume, and the zero-span tensile strength of each. In general,

these properties all showed marked changes with treatment in decrystallizing concentrations of ethylamine, but were only slightly affected above a concentration of 74%. The swollen specific volumes and freeness increased, while all other properties decreased. The specific surface showed the most continuous change with decreasing degree of crystallinity.

It was suggested that the sudden increase in swollen specific volume and the decrease in zero-span tensile strengths may have been caused by a weakening of bonds within the macrostructure of the fibers, which allowed greater swelling in water and which tended to weaken the entire fiber in a zero-span tensile test.

It would appear that the decrease in pulp surface areas was due to loss of fine material through the wire cloth at the bottom of the treatment vessel. The amount lost was apparently dependent upon ethylamine concentrations greater than 71%.

Handsheets were prepared from each of the five pulps and were pressed to dryness at 50, 250, and 1000 p.s.i. The tensile strengths and apparent elastic moduli of each were determined as a function of both crystallinity index and wet pressure. These properties were observed to undergo abrupt changes with treatment in the decrystallizing concentrations of ethylamine in a manner similar to that found for the filtration resistances and zero-span tensile strengths. The tensile strengths at 50 p.s.i. wet pressure decreased from about 7.3 kg./sq.mm. for Pulps 0 and 60 to about 4.5 kg./sq.mm. for Pulps 74, 79, and 94. The apparent elastic moduli, for the same handsheets, decreased from about 870 to about 625 kg./sq.mm.

It was suggested that these changes were caused partly by the decrease in specific surface areas resulting in decreased fiber-to-fiber bonding, and partly by the loss of intrinsic fiber strength.

The tensile creep properties of the handsheets, prepared from all five pulps and pressed at 50, 250, and 1000 p.s.i., were studied using part of the equipment designed by Brezinski (4). Special lightweight jaws were constructed for holding the specimens during testing. These jaws allowed more precise measurements of creep recovery behavior.

It was found that the 50-p.s.i. handsheets from Pulps 74, 79, and 94 exhibited considerably greater creep, at the same level of apparent initial stress, than did those from Pulps 0 and 60. As the wet pressure was increased to 50 and then to 1000 p.s.i., the difference between Pulps 0 and 74 continually decreased. However, that between Pulps 74 and 94 first decreased and then increased at 1000 p.s.i. This latter behavior was taken as evidence that the intrafiber properties of the pulps contributed to the response of paper to stress, but that the contribution did not become apparent until differences in fiber-to-fiber bonding were substantially reduced. Apparently, the differences in interfiber bonding between Pulps 0 and 74 were still great enough at 1000 p.s.i. wet pressure to overshadow the differences in intrafiber structure. Similar, but more apparent evidence, was obtained from the recovery curves of these handsheets tested under comparable conditions.

It was shown that the 50-p.s.i. handsheets from Pulp 0 followed the concept of the master creep curve described by Brezinski (4) and

Schulz (38). The shift in log time necessary to form the curve was found to be a linear function of the apparent initial stress. However, it was also found that master creep curves could not be formed from the creep curves of Pulps 74 and 94 as the creep tended to be too high at high stresses and at relatively long times. This was interpreted as evidence that the reduction in fiber crystallinity caused a decrease in the nonrecoverable portion of the first creep.

The relationship between first-creep deformation in 24 hours and first-recovery deformation, in the same period of time, was studied as a function of wet pressure for Pulps 0, 74, and 94. Three separate curves were found, one for each of the three pulps. This was compared to the results reported by Schulz (38) and Brezinski (4) who showed that wide variations in interfiber properties, such as fiber-to-fiber bonding and degree of wet-straining, had no significant effect upon this function. In a similar manner, for each of the three pulps studied here, variations in stress and interfiber bonding did not alter the position or shape of the creep-recovery curve. However, between the three pulps, as the severity of the decrystallizing treatment was increased, the amount of recovery increased (the amount of nonrecoverable creep decreased). This was taken as strong evidence that intrafiber, molecular mechanisms are important in the response of paper to tensile stress, and that these mechanisms are exhibited as a change in the paper's recoverability at equivalent levels of first-creep deformation. It was suggested that this increased recoverability with increasing ethylamine concentration may be caused by a reduction in the ability

of the fibers to form metastable, molecular structures and by an increase in their ability to form reversible structures while under the influence of stress.

In general, it appears that both inter- and intrafiber properties are important to the behavior of paper under tensile stress, and that the relative importance of each depends upon the over-all strength and structure of the sheet. As the extent of fiber-fiber bonding is increased, more of the strain response is from that of the fibers, and their properties play a greater role in determining the behavior of the specimen. Conversely, in the relatively weak sheet, almost the entire straining results from deformation and rupture of the bonds, and consequently their strength and structure become controlling.

In view of the relatively large changes in creep and recovery behavior caused by variations in fiber-to-fiber bonding, and in view of the evidence for the rupture of such bonding under tensile stress, as presented by Sanborn (19), and Page, et al. (30-32), it also appears that the major control of creep and recovery response in average-strength papers is vested in the interfiber properties. However, at least a partial contribution of fiber properties must also be considered.

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APPENDIX I

EFFECTS OF BLOTTER MOISTURE CONTENT ON CREEP BEHAVIOR

In the course of this study, it was discovered that the creep behavior of handsheets prepared under different conditions of room relative humidity could be markedly different. It was concluded that this was possibly caused by differences in the initial moisture contents of the blotters used in the wet-pressing procedure, resulting in variations in the final degree of fiber-to-fiber bonding within the handsheets. This hypothesis was investigated in the following manner.

Blotters were conditioned at relative humidities of 50 and 85% and were used in the preparation of handsheets from Pulps 0 and 94 at wet pressures of 50 and 1000 p.s.i. The usual handsheet wet-pressing procedure was used. The blotters were stored in tightly closed polyethylene bags until needed. After preparation and pressing, all sheets were conditioned at 50% R.H. and 73°F.

Creep tests were conducted for 24-hr. periods on specimens cut from each handsheet at an apparent initial stress of 3.00 kg./sq.mm. The results of these tests are shown in Fig. 27 and 28. It can be seen that the sheets prepared with blotters conditioned at 85% R.H. displayed significantly higher levels of creep than those using the 50% R.H. blotters. As expected, the differences at 1000 p.s.i. were less than those at 50 p.s.i. The total first-creep strains in 24 hours for each test are shown in Table VIII along with the percentage increases caused by the use of the 85% R.H. blotters.

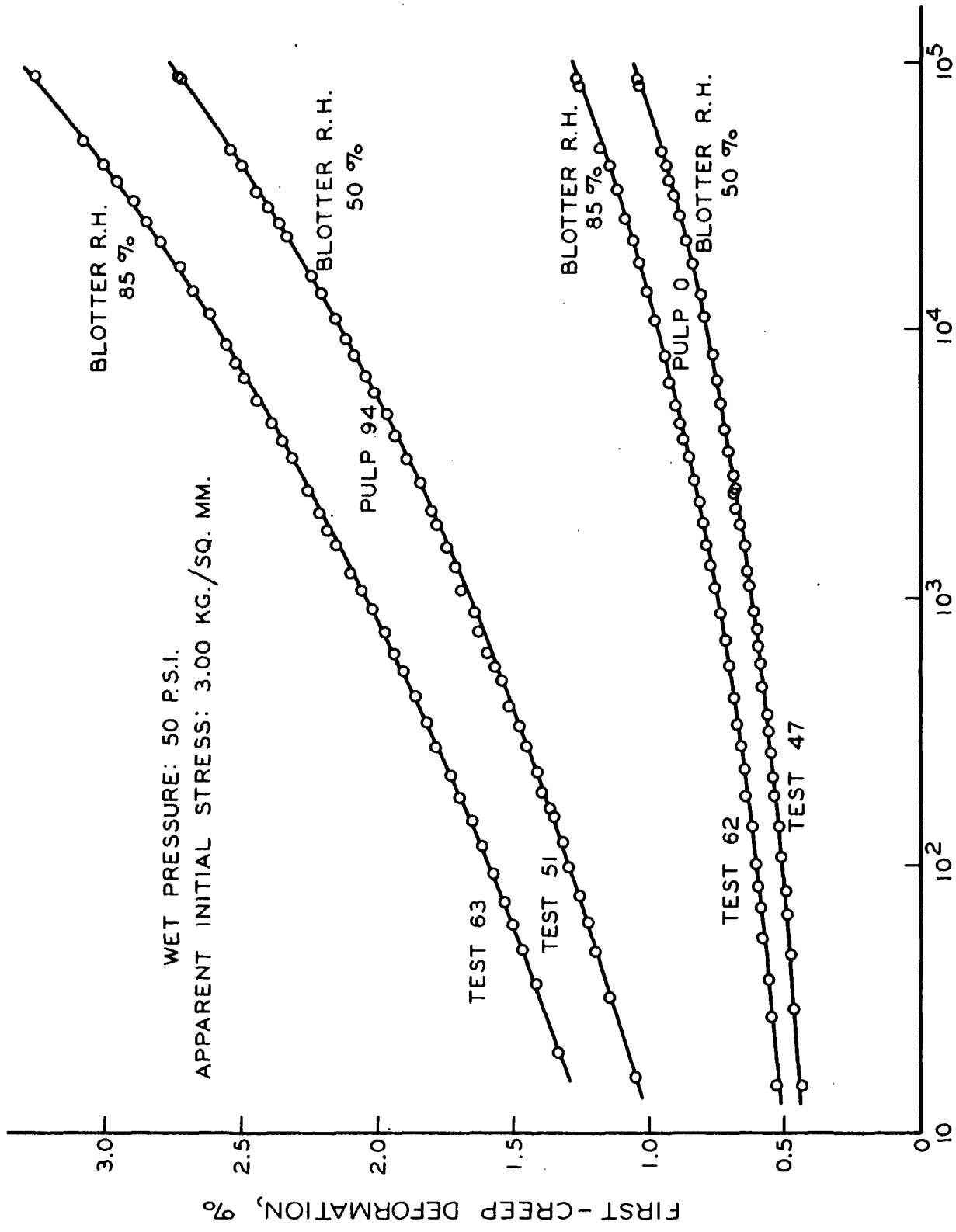

Figure 27. The Effect of Blotter-Conditioning Relative Humidity on Handsheet First-Creep Behavior

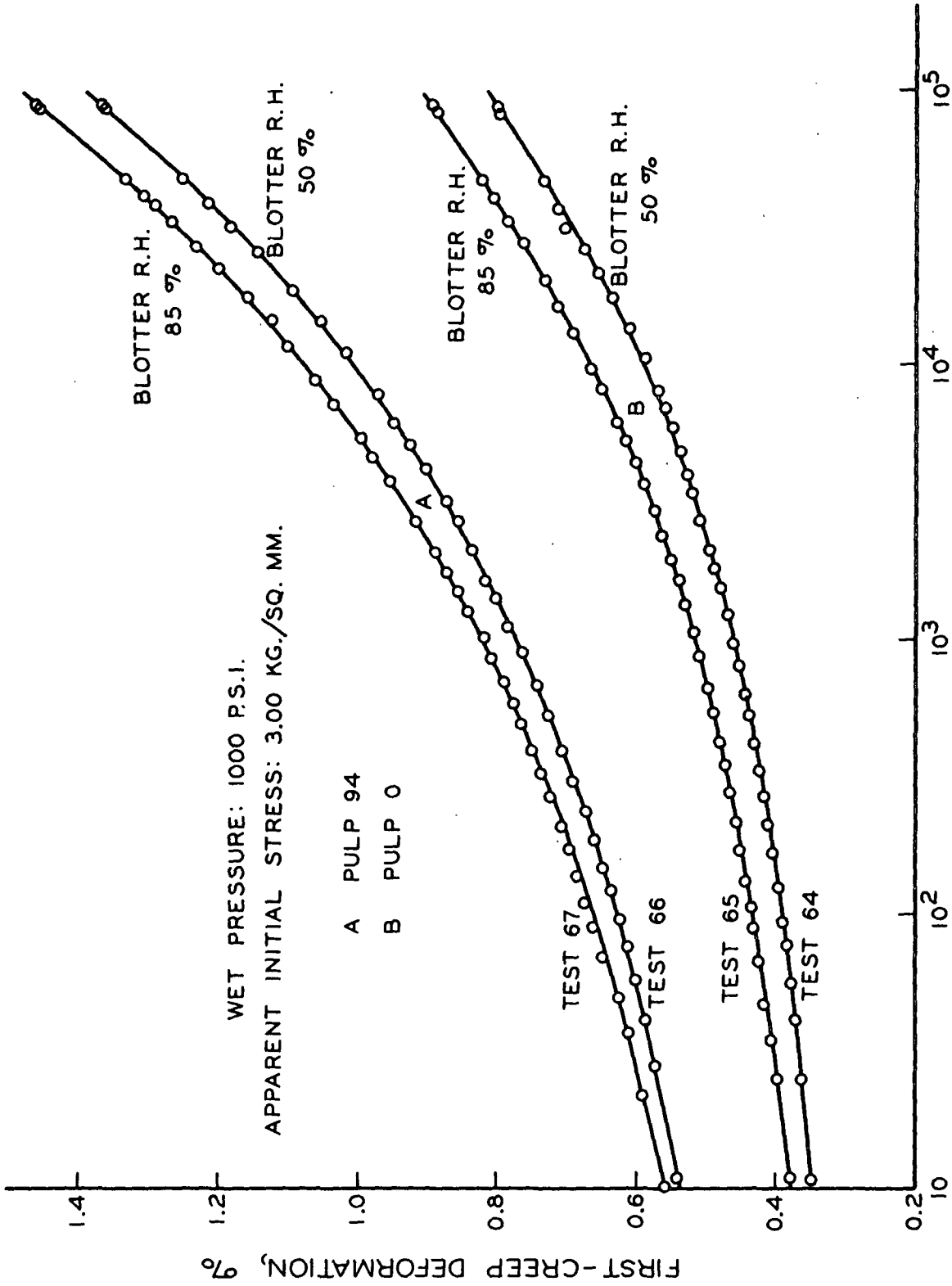

Figure 28. The Effect of Blotter-Conditioning Relative Humidity on Handsheet First-Creep Behavior

TABLE VIII

TOTAL CREEP STRAINS IN 24 HOURS OF SPECIMENS PREPARED USING BLOTTERS CONDITIONED AT 50% AND 85% R.H.

Wet Pressure, p.s.i.	Total Creep Strain, % 50% R.H.	85% R.H.	Increase, %
	Pulp O		
50	1.047	1.267	21.4
1000	0.800	0.890	11.2
	Pulp 94		
50	2.726	3.257	19.5
1000	1.367	1.461	6.9

It is believed that this behavior reflected a decrease in the extent of handsheet interfiber bonding, with increased blotter moisture content, which was caused by a lower rate of water removal in a fixed period of time, in the wet-pressing procedure. No significant differences were found in the moisture contents of the handsheets after the final conditioning at 73°F. and 50% R.H.

Further investigation of this phenomenon might prove fruitful. It was not within the scope of this thesis to conduct additional experimentation, but merely to point out the existence of the problem. In the main body of this work, absolute comparisons of creep and recovery behavior were made only with specimens which had been prepared under comparable conditions.

APPENDIX II

TIME - DEFORMATION DATA IN CREEP AND RECOVERY TESTS

In the following table, the time, in seconds, and the deformation, in mils, are taken from the instant of application or removal of the load. All deformations, in per cent, were calculated using the span length existing at the beginning of the individual creep or recovery test. The span length prior to all first-creep tests was 7.00 in.

The following information is given for each test:

Specimen number, as indicated.

B.W.: average ovendry basis weight of 1 by 8 in. test specimen, g./sq.m.

Load: total applied load, kg., including the weight of the lower jaw assembly (0.08 kg.).

AIS: apparent initial stress, kg./sq.mm., calculated assuming a cellulose density of 1.55 g./cc.

BCRH (Tests 47-60, 62-67 only): Blotter-conditioning relative humidity, %; relative humidity at which blotters were conditioned prior to use in hand-sheet preparation.

In all recovery tests, the specimen supported the weight of the lower jaw assembly.

All tests were run at a nominal temperature of 73°F. and a relative humidity of 50%.

Test No. 5

74-50-5-6
B.W.: 81.0
Load: 1.46
AIS: 1.10

First Creep

Seconds	Mils
14	12.0
30	12.1
45	12.4
51	12.5
66	12.5
87	12.7
106	12.7
120	12.8
144	12.8
170	12.9
200	12.9
240	13.0
280	13.1
332	13.2
408	13.3
432	13.4
498	13.5
570	13.5
618	13.6
695	13.6
760	13.7
970	13.8
1080	13.9
1280	13.9
1480	14.0
1900	14.2
2365	14.3
2750	14.4
3580	14.5
4310	14.6
5660	14.8
7220	15.0
9260	15.2
14,300	15.8
17,600	15.8
21,100	16.1
24,500	16.5
28,700	16.8
36,000	17.0
41,600	17.4
48,500	17.6
86,400	18.9

First Recovery

Seconds	Mils
20	11.5
37	11.4
60	11.4
100	11.4
142	11.4
166	11.5
185	11.6
233	11.8
257	11.9
352	12.1
405	12.2
450	12.3
561	12.4
692	12.5
852	12.5
1055	12.7
1320	12.9
1640	13.4
1860	12.9
2440	13.1
2680	13.1
3160	13.3
3670	13.5
4300	13.7
5170	13.8
5630	13.7
6100	13.7
7820	13.8
8700	13.9
9800	13.9
14,300	14.1
16,800	14.3
17,800	14.3
19,300	14.4
24,500	14.5
28,700	14.9
30,200	14.8
35,500	14.7
44,000	14.9
85,800	14.9
86,400	14.8

Test No. 6

74-50-5-3
B.W.: 81.4
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
11	20.9
29	21.9
41	22.2
52	22.5
70	22.7
80	22.9
93	23.0
115	23.4
130	23.5
150	23.6
189	23.9
240	24.3
282	24.5
340	24.7
398	24.9
462	25.2
570	25.5
680	25.8
830	26.2
942	26.4
1110	26.7
1350	27.1
1560	27.4
1830	27.7
2120	28.0
2480	28.4
2950	28.8
3710	29.4
4310	29.8
5570	30.5
7180	31.3
8730	32.0
10,400	32.7
13,200	33.5
20,500	35.9
25,000	35.9
33,100	37.0
68,400	41.1
69,800	41.3
75,600	41.4
80,900	41.7
85,400	42.0
86,200	42.3
86,400	42.4

Test No. 6 (Cont.)

First Recovery

Seconds	Mils
12	18.0
27	18.5
40	18.6
56	18.8
74	18.9
109	19.4
127	19.5
157	19.6
191	19.8
233	19.9
276	20.1
352	20.4
420	20.5
485	20.6
660	21.0
811	21.3
979	21.6
1250	21.7
1440	21.8
2100	22.2
2550	22.5
3220	22.8
3890	22.9
5300	23.6
5930	23.8
6790	23.9
9250	24.3
13,500	24.7
14,800	24.9
19,100	25.2
23,500	25.4
28,600	25.8
70,600	26.5
74,300	26.6
77,000	26.6
82,100	26.9
86,100	26.9
86,400	26.9

Test No. 7

74-250-5-7
B.W.: 81.1
Load: 4.54
AIS: 3.47

First Creep

Seconds	Mils
18	51.4
33	54.5
46	56.2
64	58.0
89	59.8
113	61.2
145	62.9
174	64.1
210	65.5
260	67.2
352	69.8
381	70.6
448	71.8
519	73.0
623	74.5
732	75.9
855	77.2
985	78.5
1230	80.5
1460	81.8
1730	83.1
2033	84.6
2240	85.4
2980	88.4
3465	90.1
3900	91.7
4740	94.1
5650	96.5
6480	98.2
7790	100.8
12,100	106.2
14,700	108.9
15,800	110.2
16,400	110.2
17,200	110.7
22,000	114.5
26,700	117.0
27,900	117.7
33,500	121.0
41,800	124.1
76,700	135.3
86,000	135.8
86,300	135.9
86,400	135.9

First Recovery

Seconds	Mils
15	34.0
33	35.4
46	36.2
63	36.8
77	37.2
93	37.6
116	38.0
132	38.3
151	38.7
177	39.0
204	39.3
245	39.9
264	40.1
310	40.6
343	40.8
383	41.1
425	41.4
473	41.8
546	42.1
600	42.5
700	43.0
822	43.1
918	43.9
1025	44.2
1262	45.0
1513	45.7
1825	46.2
2294	46.9
2488	47.1
3350	48.0
3675	48.2
4835	49.3
6230	50.0
8540	50.9
9730	51.3
11,000	52.1
16,000	52.7
17,400	53.0

Test No. 7 (Cont.)

Seconds	Mils
19,900	53.3
26,300	54.3
28,300	54.7
37,300	55.4
46,000	55.9
85,000	57.7
86,400	57.8

Test No. 8

84-50-5-5
B.W.: 80.8
Load: 3.99
AIS: 3.02

First Creep

Seconds	Mils
18	61.2
31	64.7
45	66.4
63	68.6
87	70.8
110	72.5
131	74.0
145	74.9
168	76.1
205	77.8
259	79.8
291	80.9
366	82.7
403	83.6
487	85.3
567	86.8
647	88.0
712	88.9
792	89.8
1123	92.3
1180	98.5
1330	94.9
1640	97.3
1860	98.4
2180	100.4
2530	102.2
3050	104.4
4200	107.5
5030	109.8
6000	112.1
6900	113.6
7800	115.2
10,300	118.8
12,000	120.9
13,000	121.9
18,800	128.0
21,800	130.3
26,800	133.3
69,100	149.5
74,000	151.0
80,200	152.3
84,300	153.3
85,700	153.5
86,400	153.9

First Recovery

Seconds	Mils
18	39.0
32	40.3
46	41.0
60	41.6
73	42.0
87	42.4
101	42.7
126	43.2
151	43.6
172	44.0
213	44.6
248	44.8
282	45.4
328	45.8
385	46.0
423	46.6
516	47.3
608	47.5
787	48.2
967	49.1
1218	49.7
1490	50.5
1928	51.4
2357	52.1
2952	52.7
3700	53.5
4810	54.5
5705	55.1
6760	55.6
8330	56.4
9780	56.8
11,400	57.4
13,400	57.9
17,800	58.8
22,900	59.7
31,200	60.4
70,300	62.8
75,200	62.7
97,300	63.7

Test No. 9

74-50-5-7
B.W.: 80.9
Load: 4.66
AIS: 3.50

First Creep

Seconds	Mils
20	100.9
39	108.2
52	113.7
65	115.2
77	117.0
90	119.1
101	120.7
119	122.6
138	124.7
164	127.2
189	129.0
221	131.1
250	133.1
311	136.9
336	138.5
386	140.7
440	142.6
492	144.3
569	146.4
642	148.1
705	149.7
820	152.2
905	153.7
990	154.9
1140	157.6
1410	161.8
1810	166.2
2200	169.5
2870	173.8
3700	178.4

Test No. 9 (Cont.)

Seconds	Mils
4770	183.4
6120	188.2
7230	191.2
10,050	197.3
12,100	200.8
13,500	203.2
14,300	204.5
17,200	208.1
19,200	210.8
22,000	213.9
23,600	215.4
25,600	216.9
30,100	220.0
34,600	222.9
43,400	227.6
82,600	242.7
86,100	243.7
86,400	243.8

First Recovery

Seconds	Mils
19	46.3
35	48.0
50	48.8
63	49.4
77	50.0
92	50.6
106	50.9
128	51.3
156	52.1
183	52.7
220	53.2
288	54.3
348	55.0
439	56.0
588	57.2
819	58.4
940	59.3
1170	60.0
1860	62.1
2720	63.6
3600	64.7
5290	66.3
10,100	69.2
13,500	70.7
14,200	70.9
17,900	71.8
23,200	72.5
32,200	73.8
43,600	74.9
85,400	77.3
86,400	77.3

Test No. 10

74-50-5-2
B.W.: 81.8
Load: 4.02
AIS: 3.00

First Creep

Seconds	Mils
13	59.5
40	66.0
58	68.4
73	70.4
94	72.4
110	73.6
125	74.6
148	76.0
172	77.2
198	78.3
248	79.6
268	80.5
300	81.5
345	82.9
392	84.0
450	85.2
493	86.1
550	87.2
620	88.3
680	89.2
770	90.3
845	91.3
960	92.6
1110	94.2
1380	96.6
1700	99.1
1940	100.4
2290	102.2
2900	104.5
3510	106.6
4250	108.8
5390	111.5
6080	113.2
7430	115.6
8600	117.6
10,200	121.1
11,100	122.4
13,500	125.5
16,300	128.3
18,700	130.4
24,200	133.7
28,300	136.2
34,700	139.1
42,900	141.7
46,800	143.0
83,100	152.2
86,400	153.2

First Recovery

Seconds	Mils
15	39.1
32	40.3
46	41.2
65	42.0
77	42.5
92	42.8
108	43.2
130	43.6
157	44.0
190	44.6
220	44.9
283	45.7
347	46.1
440	46.9
543	47.5
653	47.9
805	48.7
1005	49.1
1223	49.8
1530	50.5
1820	51.0
2180	51.7
2600	52.2
2980	52.7
4000	53.7
4910	54.3
5920	54.9
7370	55.7
8640	56.2

Test No. 10 (Cont.)

Seconds	Mils
9900	56.8
13,200	57.7
16,300	58.4
20,600	59.5
25,700	59.9
34,100	60.9
38,600	61.9
49,300	62.3
83,100	64.0
85,100	64.1
86,400	64.2

Test No. 11

74-50-4-6
B.W.: 81.1
Load: 5.58
AIS: 4.20

First Creep

Seconds	Mils
18	212.1
36	234.7
51	246.7
62	256.6
85	Rupture

Test No. 16

94-50-4-4
B.W.: 81.1
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
19	25.3
36	25.7
58	26.5
70	26.7
87	26.9
100	27.2
120	27.4
140	27.6
160	27.7
200	28.0
245	28.4
283	28.6
345	28.9
400	29.3
475	29.5
540	29.7
625	30.0
710	30.2
815	30.4
950	30.7
1070	30.8
1220	31.1
1440	31.4
1620	31.5
1870	31.8
2270	32.3
2700	32.6
3210	33.0
3710	33.4
4780	33.9
5660	34.3
6490	34.7
7410	34.9
8580	35.3
10,300	35.6
12,200	36.1
15,700	36.7
17,900	37.4
21,100	37.4
25,500	38.2
30,200	38.9
35,700	39.8
45,500	40.4
85,100	45.2
86,400	45.3

First Recovery

Seconds	Mils
13	23.0
28	23.7
43	24.0
60	24.2
76	24.5
95	24.6
113	24.7
136	24.8
167	25.0
193	25.1
240	25.4
273	25.6
331	25.7
385	25.8
446	26.0
509	26.1
600	26.4
731	26.7
893	26.9
1050	27.1
1234	27.4
1520	27.7
1920	27.9
2150	28.0
2620	28.0
3230	28.1
3630	28.5
4480	28.9
5250	29.2
6570	29.6
8040	29.9
10,000	30.6
12,100	30.5
14,900	30.9
18,000	31.1
22,000	31.5
30,100	31.8
41,000	32.5
45,600	32.8
51,200	32.6
86,400	33.1
95,300	33.4

Test No. 17

94-50-4-5
B.W.: 81.1
Load: 3.99
AIS: 3.00

First Creep

Seconds	Mils
12	62.5
28	66.4
40	68.9
51	70.8
61	72.1
72	73.1
83	74.3
94	75.1
111	76.5
124	77.4
143	78.5
165	79.9
186	80.8
210	82.1
234	83.9
285	84.6
338	86.3
398	88.0
425	88.5
490	89.9
550	91.1
672	93.0
827	95.2
945	96.8
1110	98.5
1460	101.6
1880	104.5
2290	107.0
2735	108.5
3340	110.8
3950	112.7
4710	114.8
5660	116.7
6460	118.3
7850	120.8
9130	123.1
11,400	126.3
14,100	129.7
19,300	134.9
24,200	138.7
29,300	140.4
68,800	160.1
75,400	162.3
81,800	163.5
85,100	164.6
86,400	166.9

First Recovery

Seconds	Mils
18	40.6
32	42.3
46	42.9
58	43.6
69	44.0
83	44.5
105	45.1
121	45.5
140	45.8
168	46.4
197	46.7
234	47.1
278	47.5
357	48.5
416	49.2
522	49.8
700	50.8
846	51.7
980	52.0
1100	52.6
1490	53.8
1840	54.7
2380	55.6
3220	56.7
4190	57.6
5600	58.8
7410	59.6
9200	60.6
13,700	62.3
20,000	63.7
24,700	64.5
29,400	65.3
34,900	65.6
70,000	67.6
75,400	68.4
76,100	69.0
86,000	69.1
86,400	69.2

Test No. 18

94-50-4-6
B.W.: 81.0
Load: 4.65
AIS: 3.50

First Creep

Seconds	Mils
20	122.5
40	132.6
53	137.4
68	141.8
81	145.5
96	148.4
108	150.9
128	154.2
150	156.9
175	160.1
201	163.3
252	169.0
282	171.8
310	Rupture

Test No. 19

0-50-7-3
B.W.: 81.7
Load: 2.41
AIS: 1.80

First Creep

Seconds	Mils
11	14.4
28	14.6
41	14.8
50	14.9
63	15.1
81	15.2
96	15.3
122	15.5
151	15.6
196	15.6
265	15.7
330	16.0
450	16.2
550	16.4
730	16.5
940	16.5
1250	16.7
1650	16.9
2420	17.4
3080	17.5
3910	17.7
4950	18.0
5900	18.2
7010	18.4
8080	18.6
10,800	18.9
13,600	19.4
17,900	19.8
21,500	20.2
26,000	20.6
32,200	20.7
37,100	21.0
41,600	21.2
47,200	21.5
82,000	23.3
85,000	23.3
86,400	23.3

First Recovery

Seconds	Mils
11	13.0
30	13.5
44	13.7
59	13.8
74	13.8
96	13.9
128	14.0
167	14.1
192	14.2
240	14.3
335	14.4
432	14.5
535	14.6
675	14.7
865	14.7
1040	14.9
1280	15.0
1630	15.3
2070	15.5
2500	15.6
3120	15.7
3800	15.9
4710	16.0
5680	16.2
6880	16.3
7980	16.4
11,200	16.5
14,600	16.7
18,100	16.9
21,700	17.0
25,600	17.0
35,100	17.0
40,300	17.0
42,400	17.0
82,400	17.6
84,700	17.7
86,400	17.8

Test No. 20

0-50-7-5
B.W.: 82.2
Load: 4.04
AIS: 3.00

First Creep

Seconds	Mils
9	29.2
22	30.7
39	31.6
51	32.1
65	32.5
81	32.8
100	33.4
116	33.7
145	34.3
167	34.5
192	34.8
221	35.2
272	35.7
315	36.2
378	36.6
482	37.4
586	37.8
712	38.5
888	39.2
1050	39.7
1230	40.3
1530	41.1
1840	41.7
2200	42.4
2490	42.8
2730	43.2
3410	44.1
3910	44.6
4500	45.3
5510	46.2
6710	46.8
7700	47.5
8590	47.9
10,700	48.9
14,100	50.4
17,300	51.5
20,900	

Test No. 20 (Cont.)

First Recovery

Seconds	Mils
12	24.1
27	24.9
40	25.3
53	25.7
68	25.9
78	26.1
98	26.3
123	26.7
147	27.0
169	27.1
199	27.3
235	27.6
296	27.9
382	28.2
453	28.6
537	28.9
643	29.1
777	29.4
930	29.7
1230	30.1
1520	30.4
1880	30.8
2310	31.1
2910	31.7
3640	31.9
4570	32.3
5610	32.7
6830	33.1
8340	33.7
9480	34.0
12,000	34.6
15,500	34.8
20,300	35.1
27,200	35.6
35,900	35.8
38,700	36.0
47,500	36.4
84,100	37.1
86,400	37.1

Test No. 21

0-50-7-4
B.W.: 82.0
Load: 5.64
AIS: 4.20

First Creep

Seconds	Mils
11	62.4
27	67.7
41	70.7
51	72.2
65	74.1
81	75.8
91	76.9
106	78.0
127	79.5
165	82.0
191	83.5
219	85.0
270	87.3
326	89.3
394	91.1
479	92.7
550	94.2
648	94.6
755	97.1
890	98.7
1030	100.3
1250	102.4
1350	103.1
1850	106.2
2250	108.4
2720	110.7
3400	113.5
4000	115.2
5130	117.8
6050	119.6
7110	121.4
8380	123.5
9850	124.9
11,000	126.1
13,400	127.6
17,000	130.4
21,700	133.2
28,600	137.2
37,300	141.1
39,800	142.1
48,900	144.4
85,700	153.3
86,400	153.3

First Recovery

Seconds	Mils
20	35.9
32	37.0
42	37.5
55	37.9
70	38.3
82	39.0
117	39.7
136	39.9
154	40.3
184	40.8
222	41.4
279	42.1
324	42.7
389	43.1
461	43.8
523	44.1
620	44.6
742	45.2
880	45.7
1040	46.1
1290	46.8
1640	47.3
2100	48.6
2450	48.9
2760	49.0
3560	50.0
4420	50.6
5250	51.2
6230	51.7
7520	52.1
8460	52.9
10,600	53.6
12,800	54.0
16,300	54.8
20,900	55.9
23,900	56.2
30,500	56.8
35,900	57.0
41,900	57.5

Test No. 21 (Cont.)

Seconds	Mils
49,000	57.9
85,600	59.1
86,400	59.6

Test No. 22

0-50-7-6
B.W.: 81.9
Load: 7.25
AIS: 5.40

First Creep

Seconds	Mils
13	148.7
26	158.6
40	166.2
50	170.2
61	173.7
81	178.4
98	181.4
110	183.5
141	187.6
169	190.6
188	192.6
221	195.7
252	197.7
306	200.9
366	203.7
422	205.7
500	208.5
630	212.2
738	214.7
850	217.1
1040	220.7
1230	223.0
1480	226.0
1680	227.8
2170	232.4
2610	234.3
3190	237.3
3870	240.5
4800	243.7
5490	246.1
7040	249.4
8810	252.7
10,000	254.5
13,300	258.6
17,900	262.8
21,100	265.3
27,500	270.7
33,200	274.4
38,900	277.2
46,200	280.3
82,600	292.1
85,200	292.5
86,400	292.8

First Recovery

Seconds	Mils
11	45.3
25	48.3
36	49.4
49	50.5
63	51.4
76	52.2
91	52.8
108	53.3
122	53.9
147	54.4
171	55.0
202	55.5
253	56.3
298	57.0
379	58.0
450	58.5
540	59.3
651	60.0
786	60.8
913	61.3
1220	62.4
1580	63.5
2020	64.4
2490	65.3
3230	66.3
3880	67.2
4750	68.1
5630	68.6
6200	69.1
7220	69.6
8240	70.1
9530	70.5
11,500	71.3
13,400	72.0
15,800	72.6
19,000	73.4
22,800	74.3
25,900	74.8
32,500	75.6
45,700	77.1
83,100	79.2
86,400	79.4

Test No. 23

94-50-4-2
B.W.: 81.1
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
12	23.0
30	23.7
41	24.0
53	24.2
65	24.4
78	24.6
98	24.9
129	25.1
164	25.4
193	25.6
225	25.8
300	26.1
392	26.5
485	26.8
582	27.0

Test No. 23 (Cont.)

Seconds	Mils
688	27.3
820	27.7
960	27.9
1200	28.3
1520	28.7
1830	28.9
2120	29.4
2540	29.6
2990	29.8
3710	30.5
4560	30.9
5490	31.4
6440	31.8
8100	32.4
9960	32.9
11,800	33.5
16,100	34.4
19,300	35.0
25,700	36.8
31,400	38.0
37,100	38.6
44,400	39.6
80,900	43.9
83,500	43.9
86,400	43.9

First Recovery

Seconds	Mils
13	20.1
30	20.7
49	21.0
64	21.1
83	21.2
100	21.3
131	21.4
164	21.6
203	21.9
247	22.1
327	22.3
402	22.5
480	22.8
573	23.0
645	23.1
788	23.3
913	23.4
1143	23.9
1450	24.1
1750	24.3
2100	24.5
2470	24.8
2880	25.1
3560	25.3
4410	25.5
5370	25.9
6300	26.1
7590	26.2
9780	26.6
11,500	27.0
14,000	27.2
17,200	27.4
21,000	27.4
24,100	27.5
26,800	27.7
30,700	28.0
36,700	28.5
44,000	28.5
81,500	29.1
83,600	29.4
86,400	29.2

Test No. 24

94-50-4-3
B.W.: 80.9
Load: 3.18
AIS: 2.40

First Creep

Seconds	Mils
10	36.4
22	38.3
34	39.3
47	40.2
60	40.9
73	41.4
86	42.0
100	42.4
121	43.2
147	43.9
174	44.5
201	45.1
246	45.8
287	46.4
342	47.1
413	47.7
510	48.5
602	49.3
737	50.3
885	51.1
1022	51.7
1230	52.7
1550	54.0
1920	55.2
2470	56.5
3130	57.9
3950	59.3
4750	60.4
5640	61.6
6620	62.8
7770	64.1
8900	65.1
12,000	67.5
14,100	69.1
17,700	71.4
21,500	73.0
25,300	74.3
28,300	75.3
31,000	76.1
35,100	77.2
40,900	78.3
48,400	80.3
85,400	86.0
86,400	86.4

First Recovery

Seconds	Mils
15	30.1
36	31.1
48	31.8
63	32.1
80	32.3
101	32.8
123	33.2
142	33.4
174	33.7
213	34.0
258	34.4
325	35.0
395	35.3
474	35.6
527	35.9
621	36.2
756	36.8
910	37.1

Test No. 24 (Cont.)

Seconds	Mils
1150	37.7
1440	38.1
1810	38.6
2130	39.1
2490	39.1
2690	39.3
3290	40.1
3720	40.3
4440	41.0
5080	41.2
6240	42.1
7620	42.6
9150	43.1
10,400	43.3
13,900	44.1
17,300	44.9
22,100	45.2
30,300	46.0
41,200	46.9
47,900	47.3
85,400	48.1
86,400	48.2

Test No. 25

94-50-4-7
B.W.: 81.7
Load: 4.42
AIS: 3.30

First Creep

Seconds	Mils
17	90.9
39	101.3
61	106.2
78	109.2
89	110.9
104	112.8
122	114.4
165	118.8
180	119.7
195	120.6
226	122.2
252	123.9
290	125.9
339	129.0
396	131.2
437	132.9
521	135.7
618	138.1
711	140.6
888	144.2
1030	146.2
1310	149.8
1620	153.8
1950	156.8
2390	160.5
2870	163.8
3570	168.1
4310	171.8
5320	175.3
6540	180.2
8290	184.8
10,400	188.4
15,900	197.7
20,300	202.6
26,000	208.5
33,500	214.9
70,700	233.2
73,200	233.9
80,600	236.5
85,700	237.8
86,400	238.1

First Recovery

Seconds	Mils
11	47.1
24	49.2
38	50.4
50	50.9
64	52.1
85	52.7
105	53.3
130	54.3
166	55.1
203	55.8
249	56.6
308	57.3
345	57.9
391	58.4
478	59.1
576	59.7
690	60.9
828	61.0
948	61.4
1130	62.2
1580	63.4
1930	64.2
2320	65.3
2750	65.9
3430	67.2
4330	68.0
5230	68.2
7110	67.9
8980	70.9
10,600	72.3
13,600	73.1
15,300	73.9
21,700	75.2
26,200	76.0
32,900	77.0
70,500	80.4
77,900	80.8
83,200	80.7
86,400	80.9

Test No. 27

74-250-6-6
B.W.: 81.1
Load: 3.99
AIS: 3.00

First Creep

Seconds	Mils
10	35.0
25	37.1
39	38.1
49	38.8
61	39.4
72	39.9
88	40.5
99	40.8
123	41.6
148	42.0
180	42.9
236	44.1
298	45.2
354	46.0

Test No. 27 (Cont.)

Seconds	Mils
423	46.8
507	47.6
602	48.2
758	49.1
943	50.1
1130	50.9
1440	52.0
1800	53.1
2090	53.8
2690	55.0
3150	55.9
3740	56.8
4480	57.9
5410	58.9
6420	60.0
7570	61.2
8680	62.1
10,980	63.6
13,300	65.6
16,300	67.7
19,900	69.7
24,200	71.7
30,100	74.1
38,700	76.6
44,500	77.8
48,800	78.6
85,400	84.3
86,400	84.7

First Recovery

Seconds	Mils
12	29.0
28	30.3
42	30.7
55	31.1
69	31.4
83	31.8
97	32.0
117	32.1
146	32.5
173	32.8
206	33.0
255	33.3
308	33.8
388	34.1
450	34.3
550	34.9
678	35.2
792	35.7
968	36.0
1210	36.5
1460	36.9
2020	37.5
2580	37.9
3060	38.2
4000	39.1
5080	39.9
6950	40.8
9040	41.6
11,500	41.9
14,300	42.7
16,900	43.0
21,400	43.9
24,900	44.0
29,500	44.2
36,500	44.6
42,400	45.2
47,900	45.5
86,400	46.2

Test No. 29

94-250-5-4
B.W.: 81.4
Load: 4.00
AIS: 3.00

First Creep

Seconds	Mils
14	36.8
30	38.8
41	39.7
55	40.7
66	41.4
80	41.9
92	42.5
112	43.4
132	43.9
162	44.8
196	45.5
248	46.4
321	47.5
431	48.7
494	49.2
592	50.0
736	50.9
978	52.4
1160	53.3
1550	54.7
1970	55.8
2660	57.4
3320	58.5
4170	59.8
5310	61.3
6300	62.4
7650	63.8
8800	64.9
11,500	67.4
14,000	69.0
18,800	71.7
22,100	73.3
26,700	75.4
34,200	78.3
39,200	79.3
44,900	80.6
84,100	88.4
86,400	88.6

First Recovery

Seconds	Mils
9	30.3
20	31.2
32	31.8
45	32.2
59	32.7
73	32.9
90	33.2
103	33.5
125	33.8
149	34.0
179	34.4
202	34.6
242	34.9
289	35.1
339	35.6
391	35.9
475	36.2
556	36.7
672	37.1
820	37.4
990	37.9
1140	38.2
1360	38.7
1650	39.1

Test No. 29 (Cont.)

Seconds	Mils
1950	39.6
2420	40.1
2940	40.7
3670	41.1
4810	41.9
6990	42.9
8260	43.6
11,600	44.1
13,000	44.6
16,900	45.1
21,700	45.8
27,500	46.4
35,200	46.9
40,300	47.3
47,900	47.7
86,400	48.9

Test No. 35

0-250-7-6
B.W.: 80.7
Load: 3.97
AIS: 3.00

First Creep

Seconds	Mils
10	24.3
22	25.3
33	25.6
42	25.9
55	26.4
67	26.6
85	26.8
98	27.2
111	27.4
131	27.6
171	28.0
198	28.3
228	28.6
288	29.0
318	29.3
398	29.7
470	30.0
550	30.4
651	30.7
785	31.1
915	31.5
1070	31.7
1240	32.2
1580	32.7
1830	33.2
2120	33.5
2580	34.1
3060	34.5
3610	35.1
4340	35.6
5070	36.1
6190	36.7
7450	37.5
8950	38.0
11,100	38.7
13,500	39.6
16,900	40.5
20,000	41.3
22,500	41.9
25,600	42.7
28,700	43.4
32,000	44.0
36,000	44.9
44,400	45.5
50,000	46.3
84,600	49.7
86,400	49.9

First Recovery

Seconds	Mils
10	23.1
26	24.0
43	24.3
58	24.5
74	24.8
89	25.1
108	25.3
141	25.5
176	25.7
224	26.1
296	26.4
388	26.6
483	27.0
605	27.2
742	27.5
793	27.9
1220	28.2
1560	28.5
1850	28.9
2350	29.3
3000	29.6
3840	30.0
4830	30.4
5910	30.6
7110	31.0
8060	31.2
10,600	31.6
13,400	32.0
17,200	32.3
20.800	32.5
28,300	32.8
35,200	33.2
44,300	33.4
49,700	33.5
86,200	34.5
86,400	34.6

Test No. 36

0-1000-7-5
B.W.: 81.0
Load: 3.98
AIS: 3.00

First Creep

Seconds	Mils
11	23.6
27	24.7
40	25.1
54	25.4
67	25.7
83	26.0
95	26.2
114	26.4
140	26.7
169	27.0
198	27.1
249	27.6
280	27.8
339	28.0
435	28.4
498	28.7
600	29.0
751	29.6
870	29.8
1000	30.1
1230	30.4

Test No. 36 (Cont.)

Seconds	Mils
1520	31.0
1840	31.4
2270	32.0
2650	32.3
3290	33.0
3920	33.5
4720	34.1
6070	34.9
7230	35.6
8890	36.3
12,000	37.5
15,100	38.5
17,900	39.3
22,900	40.3
26,700	40.9
30,900	41.3
36,400	42.1
70,500	46.7
86,400	47.6

First Recovery

Seconds	Mils
40	22.3
55	22.6
68	22.8
84	22.9
100	23.3
125	23.4
144	23.5
174	23.6
222	23.7
258	23.9
320	24.1
377	24.3
465	24.6
559	24.8
663	25.0
790	25.3
927	25.5
1140	25.7
1350	25.8
1520	26.0
1880	26.3
2280	26.6
2570	26.7
3060	26.9
3700	27.4
4440	27.7
5720	28.2
6660	28.5
7780	28.6
9060	28.8
10,800	29.0
14,600	29.4
21,500	29.7
26,000	30.1
30,700	30.5
36,200	30.6
36,200	30.6
71,600	31.5
72,600	31.6
76,900	31.8
83,100	32.0
86,400	31.9

Test No. 37

74-1000-6-4
B.W.: 80.6
Load: 3.96
AIS: 3.00

First Creep

Seconds	Mils
11	33.3
24	34.6
33	35.4
46	36.0
60	36.7
74	37.1
89	37.6
106	38.0
126	38.6
140	38.9
170	39.5
201	40.0
242	40.6
303	41.4
376	42.0
465	42.8
560	43.5
685	44.1
785	44.7
963	45.4
1400	46.8
1580	47.2
1870	47.8
2210	48.4
2730	49.2
3250	49.9
3980	50.7
5080	52.0
5960	52.8
7350	54.0
9080	55.4
11,800	56.8
14,400	58.0
18,200	59.8
21,800	60.8
25,500	62.0
29,200	63.4
36,100	65.8
45,200	68.1
50,900	69.6
86,400	74.7

First Recovery

Seconds	Mils
8	26.7
21	27.6
33	28.1
49	28.7
63	29.0
78	29.2
101	29.6
125	29.9
156	30.3
208	30.8
240	31.1
302	31.5
362	31.9
418	32.1
490	32.6
597	33.0
721	33.5
947	33.9
1160	34.3
1390	34.8
1780	35.2
2240	35.9
2840	36.6
3400	36.9
4110	37.2

Test No. 37 (Cont.)

Seconds	Mils
5030	37.7
6260	38.1
7250	38.3
9000	38.9
11,200	39.5
14,700	39.9
17,800	40.5
21,400	40.9
25,300	41.3
29,300	41.2
36,500	41.5
43,800	41.7
51,500	42.0
86,400	42.6

Test No. 38

94-1000-1-4
B.W.: 80.7
Load: 3.97
AIS: 3.00

First Creep

Seconds	Mils
12	37.4
26	38.7
37	39.6
50	40.4
65	41.1
83	41.6
102	42.3
119	42.7
148	43.4
186	44.2
219	44.7
310	45.9
383	46.6
495	47.6
588	48.4
698	49.1
836	49.8
1000	50.5
1240	51.5
1560	52.5

Seconds	Mils
1840	53.4
2220	54.3
2780	55.4
3550	56.5
4810	58.2
6110	59.8
7380	61.3
9350	63.1
12,900	65.7
19,700	70.0
24,300	72.2
28,900	73.9
34,700	75.8
70,100	83.1
75,400	83.9
81,400	84.4
85,000	85.0
86,400	85.3

First Recovery

Seconds	Mils
11	32.6
29	33.8
41	34.5
54	34.8
76	35.2
91	35.6
110	35.9
137	36.2
175	36.7
215	37.0
262	37.4
321	37.9
380	38.2
462	38.6
562	39.0
670	39.4
822	39.9
1040	40.4
1370	41.1
1750	41.7
2120	42.1
2690	42.7
3200	43.0
4040	43.7
5060	44.3
7080	44.9

Seconds	Mils
8930	45.6
13,100	45.8
20,200	46.9
27,500	47.7
35,300	48.2
70,000	49.4
86,400	49.6

Test No. 39

0-250-7-4
B.W.: 80.5
Load: 2.38
AIS: 1.80

First Creep

Seconds	Mils
11	12.8
27	13.1
39	13.2
56	13.4
85	13.5
105	13.6
131	13.7
161	13.8
200	13.9
253	14.0
302	14.1
360	14.2
430	14.2
520	14.3
615	14.3
920	14.5
1230	14.6
1840	14.8
2440	15.1
3050	15.3
3850	15.4
4880	15.4
6590	15.5
9160	15.8
11,200	15.8
14,300	16.1
18,200	16.3
21,400	16.4
25,200	16.6

Test No. 39 (Cont.)

Seconds	Mils
30,500	16.9
34,900	17.0
44,100	17.4
83,000	18.4
86,200	18.2
86,400	18.2

First Recovery

Seconds	Mils
17	11.5
32	11.6
54	11.7
76	11.8
105	11.9
137	12.0
187	12.1
248	12.2
320	12.3
475	12.5
585	12.6
760	12.7
1050	12.8
1630	12.9
1940	13.0
2370	13.1
2880	13.3
3610	13.5
4560	13.6
5540	13.7
6620	13.8
9400	14.0
12,200	14.1
15,800	14.5
20,300	14.5
25,200	14.6
30,700	14.2
38,900	14.6
82,300	14.8
86,400	14.9

Test No. 40

74-250-6-5
B.W.: 81.0
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
15	17.4
32	17.8
46	18.0
67	18.3
87	18.5
111	18.6
128	18.7
157	18.8
194	18.9
250	19.1
335	19.4
423	19.6
549	19.8
745	20.0
875	20.2
1040	20.4
1360	20.7
1820	20.9
2410	21.1
3040	21.4
3660	21.6
4540	21.8
5430	22.0
7040	22.3
8340	22.4
10,000	22.7
11,600	22.9
15,400	23.4
20,900	24.0
34,300	25.0
73,400	26.8
78,400	26.7
86,100	26.8
86,400	26.9

First Recovery

Seconds	Mils
9	16.6
25	17.2
34	17.5
49	17.8
64	17.8
80	17.9
99	18.0
121	18.1
157	18.2
185	18.3
222	18.4
275	18.4
365	18.6
480	19.0
609	19.1
732	19.2
900	19.2
1100	19.3
1280	19.4
1700	19.7
2000	19.9
2380	19.9
2940	20.2
3560	20.3
4210	20.4
5060	20.6
6180	20.8
7180	20.9
8970	21.0
10,500	21.0
12,800	21.1
15,300	21.1
20,900	21.1
25,000	21.4
29,000	21.5
29,600	21.6
86,400	22.0

Test No. 41

94-250-5-5
B.W.: 81.7
Load: 2.41
AIS: 1.80

First Creep

Seconds	Mils
12	17.9
30	18.3
47	18.6
65	18.8
86	18.9
110	19.0
135	19.1
181	19.3
248	19.6
290	19.7
350	19.8
456	20.0
550	20.1
720	20.3
910	20.5
1170	20.8
1440	20.9
1860	21.1
2080	21.2
2590	21.5
2970	21.7
3830	22.0
4860	22.3
5960	22.7
7420	22.8
8830	23.1
11,600	23.5
14,300	23.8
18,000	24.1
22,400	24.8
27,200	25.3
32,800	26.2
40,700	26.4
84,700	28.9
86,400	29.0

First Recovery

Seconds	Mils
10	15.7
23	16.1
37	16.2
48	16.4
64	16.5
81	16.6
97	16.7
120	16.9
147	17.0
183	17.1
220	17.2
266	17.3
333	17.4
404	17.5
488	17.6
588	17.8
685	17.9
802	18.0
977	18.2
1460	18.4
1740	18.5
2200	18.7
2640	18.9
3250	19.1
3940	19.2
4630	19.2
5510	19.4
6530	19.5
7720	19.6
8970	19.8
11,800	19.9
14,300	19.9
17,400	20.1
21,800	20.2
27,100	20.3
36,100	20.4
41,200	20.6
47,000	20.6
83,900	20.6
85,300	20.5
86,400	20.5

Test No. 42

0-1000-7-6
B.W.: 81.1
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
11	13.0
27	13.1
39	13.2
53	13.3
80	13.6
95	13.7
143	13.8
178	13.9
225	14.0
288	14.0
350	14.1
450	14.2
520	14.2
620	14.3
820	14.5
1000	14.6
1280	14.7
1560	14.8
1870	14.9
2200	15.0
2710	15.1
3370	15.2
4160	15.4
4920	15.6
5800	15.8
7210	16.1
9100	16.3
11,500	16.6
14,100	17.0
19,600	18.0
23,600	18.0
27,800	18.1
71,400	19.9
77,900	19.8
85,400	20.0
86,400	20.1

Test No. 42 (Cont.)

First Recovery

Seconds	Mils
12	13.0
23	13.1
34	13.2
49	13.3
63	13.4
84	13.5
104	13.5
126	13.6
148	13.7
194	13.8
223	13.8
266	13.9
320	13.9
410	14.0
518	14.1
658	14.1
825	14.2
984	14.3
1240	14.4
1570	14.7
1910	14.8
2350	14.9
2780	15.0
3380	15.0
4100	15.1
5100	15.2
6960	15.3
8380	15.2
13,800	15.4
22,900	15.8
27,900	15.9
33,800	15.8
77,800	16.4
85,600	16.5
86,400	16.5

Test No. 43

74-1000-6-5
B.W.: 80.7
Load: 2.38
AIS: 1.80

First Creep

Seconds	Mils
11	15.9
26	16.2
40	16.3
52	16.4
73	16.6
91	16.8
125	16.9
161	17.1
200	17.2
230	17.3
285	17.5
338	17.6
414	17.8
518	18.0
608	18.0
780	18.2
908	18.3
1010	18.4
1380	18.7
1700	18.8
1750	18.9
2210	18.9
2620	19.1
3220	19.3
4090	19.6
5280	20.1
6710	20.3
7820	20.5
10,900	21.2
13,100	21.4
16,300	21.7
20,600	22.2
26,000	22.7
35,100	23.4
40,100	23.7
45,800	24.1
82,700	26.6
86,400	26.6

First Recovery

Seconds	Mils
18	14.5
33	14.8
47	14.9
62	15.0
78	15.2
104	15.3
137	15.5
167	15.6
207	15.6
239	15.8
328	15.8
398	15.9
492	16.2
662	16.5
836	16.6
1100	16.8
1280	16.7
1610	17.2
1890	17.3
2330	17.5
2880	17.8
3520	17.9
3940	17.9
5020	18.2
6470	18.3
7830	18.5
11,200	19.0
14,100	19.3
17,700	19.4
21,300	19.6
25,800	19.7
31,200	19.7
38,000	20.0
45,200	19.8
80,700	20.2
86,500	20.3

Test No. 44

94-1000-1-5
B.W.: 80.5
Load: 2.37
AIS: 1.80

First Creep

Seconds	Mils
11	17.9
27	18.2
48	18.5
59	18.7
72	18.9

Test No. 44 (Cont.)

Seconds	Mils
87	19.0
106	19.1
130	19.3
146	19.3
165	19.4
189	19.5
220	19.6
278	19.8
328	19.9
383	20.0
430	20.0
545	20.2
650	20.2
750	20.3
880	20.3
1020	20.4
1250	20.6
1540	20.7
1740	20.8
2170	20.9
2540	20.9
3140	21.1
3800	21.2
4730	21.2
5050	21.3
5780	21.5
6760	21.7
8310	22.0
9600	22.2
13,100	22.8
16,000	23.1
19,600	23.4
23,200	23.7
26,700	24.0
33,000	24.4
39,900	24.6
47,000	25.2
82,400	26.7
86,100	27.1
86,400	27.2

First Recovery

Seconds	Mils
15	16.0
30	16.7
43	16.9
58	17.0
80	17.2
103	17.4
140	17.6
185	17.8
245	17.9
297	18.0
375	18.1
493	18.3
633	18.5
775	18.7
930	18.8
1160	18.9
1420	19.0
1820	19.2
2190	19.5
2610	19.6
3050	19.7
3740	19.9
4670	20.2
5740	20.4
6720	20.6
9120	20.6
12,600	20.8
15,100	20.9
18,800	21.0
23,000	21.2
27,500	21.2
32,800	21.3
40,400	21.7
46,800	21.8
83,500	21.9
86,400	22.0

Test No. 45

74-50-4-5
B.W.: 81.0
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
12	23.0
29	23.6
39	23.9
53	24.1
67	24.3
85	24.5
100	24.8
122	25.0
148	25.1
178	25.2
200	25.3
242	25.6
290	25.8
350	26.0
462	26.3
545	26.5
665	26.7
812	27.0
998	27.2
1230	27.4
1410	27.7
1690	27.9
1959	28.2
2420	28.6
2810	28.9
3510	29.2
4140	29.6
5280	30.1
6410	30.6
7330	30.9
8970	31.3
11,200	31.9
13,500	32.4
19,000	33.4
22,900	34.0
25,900	34.2
32,800	35.3
68,500	38.5
76,100	39.2
85,000	39.6
86,400	39.8

First Recovery

Seconds	Mils
8	20.0
20	20.6
29	20.8
44	21.1
55	21.4
72	21.6
96	21.8
120	21.9

Test No. 45 (Cont.)

Seconds	Mils
141	22.1
190	22.3
226	22.6
277	22.9
368	23.1
475	23.5
623	23.4
763	23.8
972	23.9
1280	24.3
1580	24.5
1980	24.7
2550	25.2
3280	25.5
3760	25.7
4550	25.8
5500	25.9
6720	26.2
8760	26.5
11,000	26.7
13,300	26.9
18,700	27.2
23,000	27.5
26,200	27.8
32,800	28.0
69,300	28.4
85,000	29.0
86,400	28.8

Test No. 46

0-1000-7-3
B.W.: 80.9
Load: 2.39
AIS: 1.80

First Creep

Seconds	Mils
10	12.2
25	12.5
36	12.6
49	12.8
67	12.9
81	13.0
97	13.0
126	13.1
145	13.1
172	13.2
210	13.2
259	13.3
330	13.5
393	13.6
488	13.7
610	13.7
750	13.8
910	13.9
1120	14.0
1330	14.0
1610	14.1
1950	14.1
2350	14.2
2670	14.3
3140	14.5
3530	14.6
4320	14.7
5040	14.7
6080	14.8
7420	14.9
8530	15.0
10,100	15.0
11,800	15.2
15,400	15.6
17,900	15.7
21,400	15.8
25,600	16.1
30,200	16.4
35,500	16.7
43,100	16.8
49,600	17.0
86,400	18.0

First Recovery

Seconds	Mils
8	12.1
25	12.3
41	12.5
57	12.7
68	12.8
86	12.8
105	12.9
126	12.9
155	13.0
191	13.0
257	13.1
355	13.2
475	13.3
615	13.4
754	13.6
940	13.7
1220	13.8
1550	14.0
1900	14.1
2240	14.2
2630	14.2
3070	14.3
3500	14.4
4180	14.5
5240	14.7
6280	14.8
7690	14.9
9450	14.8
12,000	14.9
16,400	14.9
23,900	14.8
31,400	14.9
39,200	15.0
50,600	15.1
86,400	15.2

Test No. 47

0-50-11-3
B.W.: 81.3
Load: 4.00
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
15	30.6
29	32.3
46	33.4
65	34.3
79	34.9
107	35.7
137	36.5
180	37.3
210	37.7

Test No. 47 (Cont.)

Seconds	Mils
260	38.5
310	39.1
360	39.6
462	40.5
560	41.2
650	41.8
762	42.5
872	43.2
1090	44.0
1240	44.9
1520	45.7
1840	46.7
2150	47.3
2450	47.8
2500	48.0
2870	48.6
3480	49.5
4230	50.5
5230	51.5
6480	52.6
8130	53.9
11,000	55.7
13,700	57.2
17,400	58.8
21,600	60.8
26,600	62.2
31,800	63.8
36,000	64.7
40,900	65.4
46,600	66.9
82,900	72.6
86,400	73.3

First Recovery

Seconds	Mils
16	24.7
36	25.5
53	25.9
73	26.2
90	26.6
109	26.8
134	27.0
170	27.4
206	27.7
258	27.9
310	28.2
389	28.6
477	28.9
596	29.4
730	29.7
940	30.3
1130	30.8
1400	31.1
1680	31.6
2040	32.0
2400	32.3
3140	32.8
3780	33.2
4480	33.8
5130	34.2
5940	34.5
6840	34.8
8080	35.1
10,400	35.7
12,600	35.8
14,200	36.3
17,900	36.8
23,000	36.9
26,300	37.1
32,200	37.4
37,200	38.1
45,600	38.5
83,400	39.5
86,400	39.1

Test No. 48

60-50-10-4
B.W.: 83.2
Load: 4.09
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
11	32.1
26	32.8
37	34.7
50	35.5
62	36.2
76	36.7
99	37.5
117	38.0
141	38.6
170	39.3
203	39.8
252	40.6
319	41.5
365	41.9
452	42.9
533	43.7
640	44.6
750	45.3
950	46.4
1270	47.9
1580	49.0
1850	50.1
2240	50.9
2540	51.6
3000	52.7
3720	54.1
4680	55.6
5480	56.7
6450	57.7
7430	58.8
8470	59.8
9620	60.9
11,000	62.0
12,600	63.0
14,600	64.2
20,200	66.8
23,800	68.3
29,000	69.6
34,900	71.6
65,200	78.8
77,900	79.5
86,400	80.5

First Recovery

Seconds	Mils
7	22.1
21	23.3
32	23.8
47	24.1
66	24.6
81	24.8
103	25.1
131	25.4

Test No. 48 (Cont.)

Seconds	Mils
169	25.7
198	26.0
250	26.4
316	26.7
385	27.0
481	27.3
600	27.7
720	28.1
880	28.5
1050	28.8
1240	29.1
1500	29.4
1820	29.9
2190	30.2
2660	30.7
3570	31.2
4470	31.8
5360	32.1
6340	32.5
8180	33.0
11,100	33.5
14,600	34.0
20,400	34.7
25,500	35.6
33,900	36.0
71,800	37.6
76,200	38.0
86,400	38.3

Test No. 51

94-50-8-5
B.W.: 82.0
Load: 4.03
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
16	73.5
32	79.7
47	83.1
62	85.4
76	87.5
97	89.7
120	91.9
150	94.1
163	95.0
187	96.6
220	98.5
277	101.0
326	103.0
384	105.4
448	107.3
524	109.3
614	111.4
727	113.6
865	114.9
1040	118.3
1280	119.9
1510	121.8
1840	124.4
2060	125.7
2610	128.9
3280	132.2
3930	134.7
4720	137.5
5750	140.5
6710	142.8
7880	145.3
9160	147.5
10,900	150.4
13,600	153.9
15,800	156.7
22,000	162.7
24,700	164.7
28,100	167.4
32,800	170.5
40,100	174.5
46,600	177.6
84,800	190.2
86,400	190.8

First Recovery

Seconds	Mils
11	39.8
24	41.4
37	42.6
50	43.2
64	43.9
78	44.2
94	44.8
111	45.2
133	45.7
158	46.2
188	46.8
243	47.6
324	48.4
405	49.2
499	50.0
611	50.8
736	51.4
935	52.2
1090	52.9
1340	53.6
1560	54.1
1930	55.0
2310	55.7
2810	56.6
3470	57.3
3960	57.9
4720	58.6
5550	59.2
6530	59.9
7670	60.6
9200	61.3
11,800	62.4
14,700	63.4
18,700	64.4
22,800	65.1
27,000	65.8
33,100	66.5
38,200	67.2
49,100	68.1
84,100	70.3
86,400	70.3

Test No. 52

74-50-8-6
B.W.: 83.0
Load: 4.08
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
10	66.8
23	73.5
34	76.4
45	78.3
56	80.1
74	82.4
88	84.0
101	85.4
113	86.4
135	87.9
154	89.3
170	90.3
196	91.7
218	93.1
260	95.1
307	96.8
351	98.1
423	100.1
490	101.5
584	103.3
664	104.6
808	106.7
922	108.5
1180	111.3
1340	112.7
1540	114.6
1770	116.5
2100	118.9
2450	120.9
2920	123.0
3320	124.5
3840	126.3
4480	128.0
5110	129.9
6070	132.2
7220	134.8
8680	137.4
10,100	139.7
12,900	143.8
15,500	146.6
19,700	150.0
23,700	152.9
28,000	155.4
33,900	158.9
39,200	160.8
50,000	165.2
85,100	173.8
86,400	174.4

First Recovery

Seconds	Mils
12	38.4
25	39.8
38	40.6
53	41.5
68	42.0
82	42.5
98	43.0
120	43.5
158	44.1
190	44.6
223	45.0
275	45.7
342	46.2
422	46.8
523	47.3
641	48.0
778	48.6
940	49.1
1180	49.6
1500	50.7
1920	51.6
2290	52.1
2810	52.9
3340	53.5
4050	54.1
4760	54.8
5680	55.5
6560	55.9
7650	56.6
8760	57.2
10,400	57.8
13,400	58.8
17,600	59.7
22,900	60.8
28,400	61.4
34,800	62.0
39,800	62.8
51,200	63.2
86,400	65.2

Test No. 54

79-50-8-5
B.W.: 82.3
Load: 4.05
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
12	69.0
29	75.6
46	79.2
57	81.0
67	82.5
79	84.0
94	85.5
108	87.0
126	88.3
141	89.4
167	91.2
198	93.0
240	95.2
296	97.7
356	99.8
410	101.3
505	103.7
617	106.1
749	108.4
912	111.0
1120	113.6
1310	115.7
1530	117.8
1830	120.4
1830	120.4
2130	122.6
2440	124.5
2900	127.9
3490	129.6
4110	132.1
4850	134.8

Test No. 54 (Cont.)

Seconds	Mils
5530	137.0
6350	139.0
7570	141.4
9030	144.3
12,300	149.1
15,200	152.4
18,300	155.6
21,700	158.5
27,100	162.8
33,400	166.7
38,500	169.3
50,000	174.8
84,700	185.2
86,400	185.4

First Recovery

Seconds	Mils
15	39.0
29	40.3
40	41.3
52	42.0
67	42.6
80	43.1
96	43.5
120	44.1
140	44.5
168	45.1
199	45.4
243	46.1
305	46.8
365	47.3
455	48.0
559	48.6
677	49.3
840	50.1
1020	50.7
1460	52.1
1750	52.7
2190	53.6
2500	54.1
3010	54.8
3700	55.5
4500	56.4
5530	57.2
6700	58.1
8630	59.2
11,900	60.3
15,300	61.3
19,800	62.4
25,900	63.2
32,000	63.9
46,700	65.3
86,400	67.6

Test No. 55

0-50-11-5
B.W.: 82.0
Load: 2.42
AIS: 1.80
BCRH: 50%

First Creep

Seconds	Mils
10	14.4
27	14.7
42	14.9
55	15.1
67	15.2
86	15.4
105	15.5
125	15.6
151	15.7
184	15.9
253	16.2
312	16.3
410	16.5
540	16.7
705	17.0
880	17.3
1060	17.5
1290	17.6
1530	17.9
1810	18.1
2190	18.4
2550	18.6
3180	18.8
3970	19.3
5120	19.6
6110	19.9
7100	20.2
8890	20.6
10,900	21.1
13,400	21.6
19,800	22.5
24,800	22.7
36,500	24.2
71,000	26.1
76,100	25.7
81,500	25.7
86,400	26.1

Test No. 57

74-50-7-3
B.W.: 82.2
Load: 2.42
AIS: 1.80
BCRH: 50%

First Creep

Seconds	Mils
14	25.2
34	26.0
45	26.3
59	26.6
82	27.0
106	27.3
132	27.7
167	27.9
219	28.4
263	28.7
337	29.1
410	29.4
488	29.7
592	30.0
720	30.3
889	30.7
1100	31.1
1430	31.6
1720	32.0

Test No. 57 (Cont.)

Seconds	Mils
2010	32.3
2430	32.5
3020	33.0
3500	33.5
4110	33.8
5040	34.5
6120	35.0
8310	36.0
10,500	36.9
13,300	37.7
17,600	39.0
20,300	39.6
27,500	41.2
35,400	42.7
47,200	44.6
83,500	48.0
86,400	48.6

Test No. 58

60-50-10-3
B.W.: 81.9
Load: 2.42
AIS: 1.80
BCRH: 50%

First Creep

Seconds	Mils
10	14.9
27	15.4
43	15.7
60	15.9
82	16.1
101	16.3
136	16.4
175	16.6
262	16.7
334	16.9
405	17.1
490	17.3
608	17.5
730	17.7
890	17.8
1160	18.0
1370	18.3
1560	18.4
1870	18.7
2220	19.0
2620	19.3
2960	19.5
3400	19.6
4260	20.0
4880	20.3
5880	20.6
7150	20.9
9200	21.4
12,200	21.8
16,400	22.6
19,200	22.8
26,300	23.8
34,300	24.6
46,000	25.5
82,300	27.4
86,400	27.7

Test No. 59

79-50-8-6
B.W.: 82.0
Load: 2.42
AIS: 1.80
BCRH: 50%

First Creep

Seconds	Mils
12	24.4
24	25.4
37	25.9
51	26.3
63	26.5
84	27.0
107	27.3
135	27.6
164	28.0
206	28.3
271	28.8
334	29.2
425	29.6
530	30.1
625	30.3
748	30.7
892	31.1
1070	31.5
1300	31.9
1520	32.3
1790	32.5
2150	33.0
2630	33.4
3210	34.0
3790	34.3
4470	34.7
5630	35.3
7140	36.1
9020	36.9
11,300	37.6
14,800	38.6
17,800	39.5
22,500	40.5
27,000	41.4
33,800	43.0
39,800	43.8
46,800	44.5
84,000	49.4
86,400	49.6

Test No. 60

94-50-8-6
B.W.: 82.3
Load: 2.43
AIS: 1.80
BCRH: 50%

First Creep

Seconds	Mils
13	25.2
29	25.8
43	26.3
62	26.7
79	27.0
105	27.3
137	27.7
210	28.3
259	28.8
325	29.3
426	29.8
510	30.2
650	30.6
795	30.9

Test No. 60 (Cont.)

Seconds	Mils
930	31.2
1160	31.7
1400	32.1
1650	32.3
1960	32.8
2380	33.2
2790	33.6
3340	34.1
3960	34.5
4810	35.0
5560	35.4
7350	36.4
9730	37.3
13,100	38.4
16,300	39.6
20,800	40.9
24,500	42.1
32,100	43.6
38,200	44.7
45,100	45.5
82,400	50.8
86,400	51.0

Test No. 61

94-1000-1-6
B.W.: 80.1
Load: 2.36
AIS: 1.80

First Creep

Seconds	Mils
12	17.9
28	18.4
40	18.5
55	18.7
72	18.9
102	19.1
131	19.3
173	19.4
220	19.5
295	19.7
365	19.8
464	20.1
590	20.3
755	20.4
940	20.5
1150	20.6
1410	20.8
1700	20.9
2100	21.0
2530	21.2
3000	21.3
3710	21.4
4560	21.6
5660	22.0
6920	22.3
8040	22.5
9800	22.7
12,500	23.1
15,600	23.5
19,700	24.0
23,200	24.2
27,900	24.4
35,500	25.1
38,900	25.4
42,400	25.9
83,000	27.2
86,400	27.2

First Recovery

Seconds	Mils
8	16.4
20	16.8
35	16.9
51	17.1
67	17.3
92	17.5
121	17.6
160	17.8
196	17.8
258	18.0
312	18.2
410	18.3
535	18.5
668	18.6
870	18.9
1110	19.0
1360	19.2
1680	19.4
1920	19.5
2350	19.7
2830	19.8
3410	19.9
4140	20.1
4810	20.2
5940	20.4
6860	20.6
8090	20.8
10,000	20.9
12,700	20.9
15,500	21.0
19,700	21.0
23,600	21.0
28,400	21.1
33,600	20.9
39,200	21.4
48,600	21.5
85,300	21.8
86,400	21.9

Test No. 62

0-50-12-3
B.W.: 82.5
Load: 4.06
AIS: 3.00
BCRH: 85%

First Creep

Seconds	Mils
15	36.6
27	38.4
37	39.3
53	40.5
69	41.4
83	42.1
100	42.6
139	43.9
180	45.0
223	45.6
275	46.6
330	47.4
418	48.5
552	49.7
683	50.7
860	52.2
1080	53.4
1300	54.4
1540	55.4

Test No. 62 (Cont.)

Seconds	Mils
1870	56.4
2230	57.4
2710	58.6
3320	60.2
3830	61.3
4400	62.2
5190	63.4
6230	64.7
8050	66.6
10,700	68.7
13,800	70.9
17,900	73.1
21,500	74.7
26,000	76.4
33,700	78.3
40,700	80.9
47,200	82.5
81,200	88.4
86,400	88.7

Test No. 63

94-50-9-5
B.W.: 82.7
Load: 4.06
AIS: 3.00
BCRH: 85%

First Creep

Seconds	Mils
20	93.2
36	99.4
48	102.6
60	105.0
72	106.9
92	109.9
118	113.0
144	115.3
178	118.2
217	121.2
276	124.4
340	127.2
420	130.1
522	133.1
608	135.4
727	138.1
895	141.3
1040	143.9
1220	146.7
1540	150.3
1770	152.5
2050	154.8
2480	157.7
2930	160.2
3210	162.0
3760	164.4
4410	167.1
5400	171.1
6440	174.0
7340	176.2
8660	179.0
11,200	183.2
13,900	187.2
17,000	190.9
21,000	195.7
25,000	199.2
29,600	203.0
35,000	207.5
40,400	210.6
50,000	215.3
86,400	228.0

Test No. 64

0-1000-11-5
B.W.: 81.3
Load: 4.00
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
11	24.3
25	25.3
41	25.9
56	26.4
77	26.8
94	27.2
126	27.7
167	28.2
210	28.7
266	29.2
331	29.6
422	30.2
531	29.6
627	31.1
800	31.7
970	32.3
1230	32.9
1510	33.6
1800	34.3
2220	34.8
2710	35.6
3410	36.5
3980	37.0
4800	37.7
5910	38.6
6900	39.3
8070	39.9
10,500	41.3
13,600	42.6
17,800	44.5
21,700	45.9
26,400	47.4
31,500	49.2
37,000	49.9
46,700	51.6
83,300	55.9
86,400	56.0

Test No. 65

0-1000-10-4
B.W.: 81.9
Load: 4.03
AIS: 3.00
BCRH: 85%

First Creep

Seconds	Mils
11	26.5
25	27.8
35	28.5
47	29.1
67	29.6
89	30.2

Test No. 65 (Cont.)

Seconds	Mils
107	30.5
131	31.0
170	31.5
215	32.0
280	32.6
355	33.2
428	33.7
524	34.2
663	34.8
870	35.7
1050	36.3
1350	37.2
1620	37.8
1910	38.5
2400	39.4
2930	40.4
3670	41.3
4420	42.3
5210	43.3
6120	44.1
8150	45.7
9650	46.6
13,100	48.5
16,200	49.9
20,500	51.4
27,700	53.5
33,100	55.1
40,300	56.4
46,800	57.7
85,000	62.1
86,400	62.3

Test No. 66

94-1000-7-4
B.W.: 81.8
Load: 4.02
AIS: 3.00
BCRH: 50%

First Creep

Seconds	Mils
11	37.9
28	39.9
41	41.0
58	42.0
76	42.9
96	43.7
122	44.6
147	45.2
185	46.1
238	47.2
308	48.4
391	49.5
525	50.8
685	52.0
900	53.6
1120	55.1
1410	56.3
1640	57.1
2100	58.5
2680	60.0
3150	61.2
4100	63.2
5040	64.8
6120	66.4
7750	68.0
11,000	71.2
14,200	73.8
18,600	76.5
25,600	80.2
31,200	82.8
38,100	85.2
47,000	87.7
83,100	95.4
86,400	95.7

Test No. 67

94-1000-6-3
B.W.: 82.0
Load: 4.03
AIS: 3.00
BCRH: 85%

First Creep

Seconds	Mils
10	38.7
22	41.3
37	42.7
50	43.6
70	45.2
90	46.3
111	47.1
139	47.8
173	48.6
210	49.4
269	50.6
329	51.5
397	52.4
490	53.4
580	54.3
700	55.2
860	56.5
1000	57.3
1260	58.9
1480	60.0
1730	61.2
2080	62.3
2660	64.2
3720	66.8
4550	68.6
5320	69.8
7030	72.5
8690	74.2
11,600	77.1
14,300	78.7
17,500	81.1
22,200	84.1
26,800	86.3
32,800	88.8
37,500	90.3
40,900	91.4
46,900	93.4
85,700	102.1
86,400	102.3

Test No. 68

0-50-7-7
B.W.: 82.1
Load: 4.84
AIS: 3.60

First Creep

Seconds	Mils
86,400	97.9

First Recovery

Seconds	Mils
86,400	49.3

Test No. 69

0-250-7-3
B.W.: 81.5
Load: 6.68
AIS: 5.00

First Creep

Seconds	Mils
86,400	190.6

First Recovery

Seconds	Mils
86,400	66.1

Test No. 70

0-250-7-7
B.W.: 81.2
Load: 7.99
AIS: 6.00

First Creep

Seconds	Mils
86,400	292.8

First Recovery

Seconds	Mils
86,400	80.7

Test No. 71

74-50-5-4
B.W.: 80.6
Load: 4.36
AIS: 3.30

First Creep

Seconds	Mils
86,400	188.2

First Recovery

Seconds	Mils
86,400	71.9

Test No. 72

74-250-6-3
B.W.: 80.4
Load: 5.00
AIS: 3.80

First Creep

Seconds	Mils
86,400	161.2

First Recovery

Seconds	Mils
86,400	65.6

Test No. 73

74-250-6-4
B.W.: 80.1
Load: 5.38
AIS: 4.10

First Creep

Seconds	Mils
86,400	210.1

First Recovery

Seconds	Mils
86,400	76.2

Test No. 74

94-50-5-3
B.W.: 82.0
Load: 4.23
AIS: 3.15

First Creep

Seconds	Mils
86,400	183.9

First Recovery

Seconds	Mils
86,400	74.1

Test No. 75

94-250-5-2
B.W.: 81.8
Load: 5.10
AIS: 3.80

First Creep

Seconds	Mils
86,400	185.4

Test No. 75 (Cont.)

First Recovery

Seconds	Mils
86,400	74.4

Test No. 76

94-250-5-3
B.W.: 81.9
Load: 5.37
AIS: 4.00

First Creep

Seconds	Mils
86,400	218.9

First Recovery

Seconds	Mils
86,400	80.4